

## **Spectra Library – Electron Capture Negative Ion (ECNI) Mass Spectra of Selected Polybrominated Diphenyl Ethers (PBDEs)**

The following 63 mass spectra were measured by gas chromatography (GC) (6890N, Agilent Tech., Palo Alto, CA) with mass spectrometer (MS) detection (JMS-GC Mate II, JEOL, Peabody, MA.) using ECNI; methane (99.99%) as the reagent gas. (The JMS-GC Mate II is a benchtop magnetic sector MS with similar ion optics as larger high-resolution double-focusing MS systems). Individual PBDE standards were obtained from third-party analytical standard manufacturers. Each PBDE standard was diluted with hexane to a final injection concentration of approximately 500 pg/ $\mu$ L, on-column. Standards were introduced (1- $\mu$ L) into the split/splitless injector, equipped with a glass liner (1 mm, ID), using a 15 m retention column (J&W Scientific, Agilent Tech., DB-5HT, 15 m x 0.25 mm, 0.1  $\mu$ m stationary phase) to separate analytes from their carrier solvent (hexane). The injector temperature was 300 °C and initial carrier gas (helium) head pressure was 50-psi. The split vent was opened and pressure reduced to 15.2 psi after 4 minutes, following sample injection. Thereafter, column flow rate (1.5 mL/min.) was kept constant throughout the remaining portion of the run, by increasing carrier gas pressure at the head of the column compensating for increasing oven temperatures. Initial column oven temperature was 90 °C, held for 4 minutes, then increased to 150 °C at 30 °C/minute, then 10 °C/minute to 300 °C, and held for 7 minutes. Then increased to 350 °C at 30 °C/minute and held at 350 °C for 5 minutes, as a column bake-out procedure.

The MS was tuned and calibrated with a 1000:1 mixture of perfluorokerosene (PFK) and 1,2,4 trichlorobenzene, using ion 331  $m/z$ , resolution 1200. Mass calibration included 84 points, ranging from 35.0 to 998  $m/z$ . Typical values for the ion source were: electron energy 200 eV, filament 300  $\mu$ A. A source temperature of 200 °C was observed optimal to produce fragmentation characteristic of PBDE ether cleavage and molecular ions for the higher brominated compounds. ECNI mass spectra were collected in full scan (10 to 1000  $m/z$ ), at 1.0 scan/sec. All reported spectra were identified at greater than one-half peak height.

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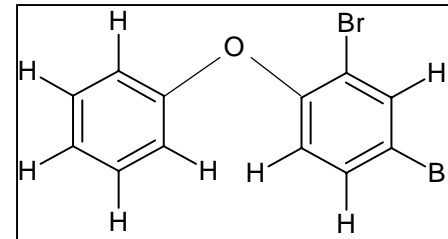
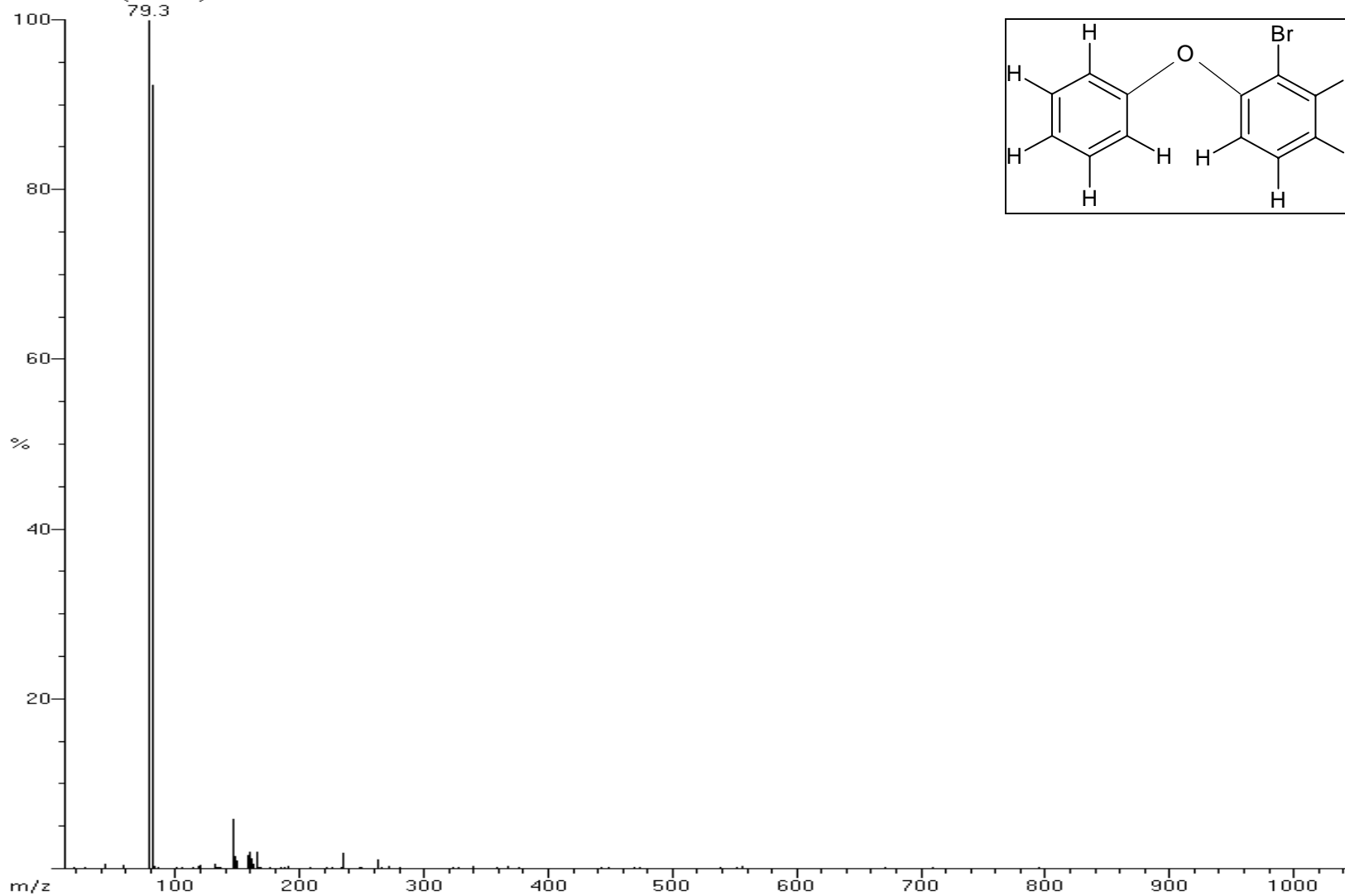
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Standard Suppliers: 1=AccuStandard, Inc. New Haven CT. USA, 2=Ultra Scientific, North Kingstown, RI. USA, 3=Wellington Laboratories, Ontario, Canada, 4=Cambridge Isotope Laboratories, Andover, MA. USA

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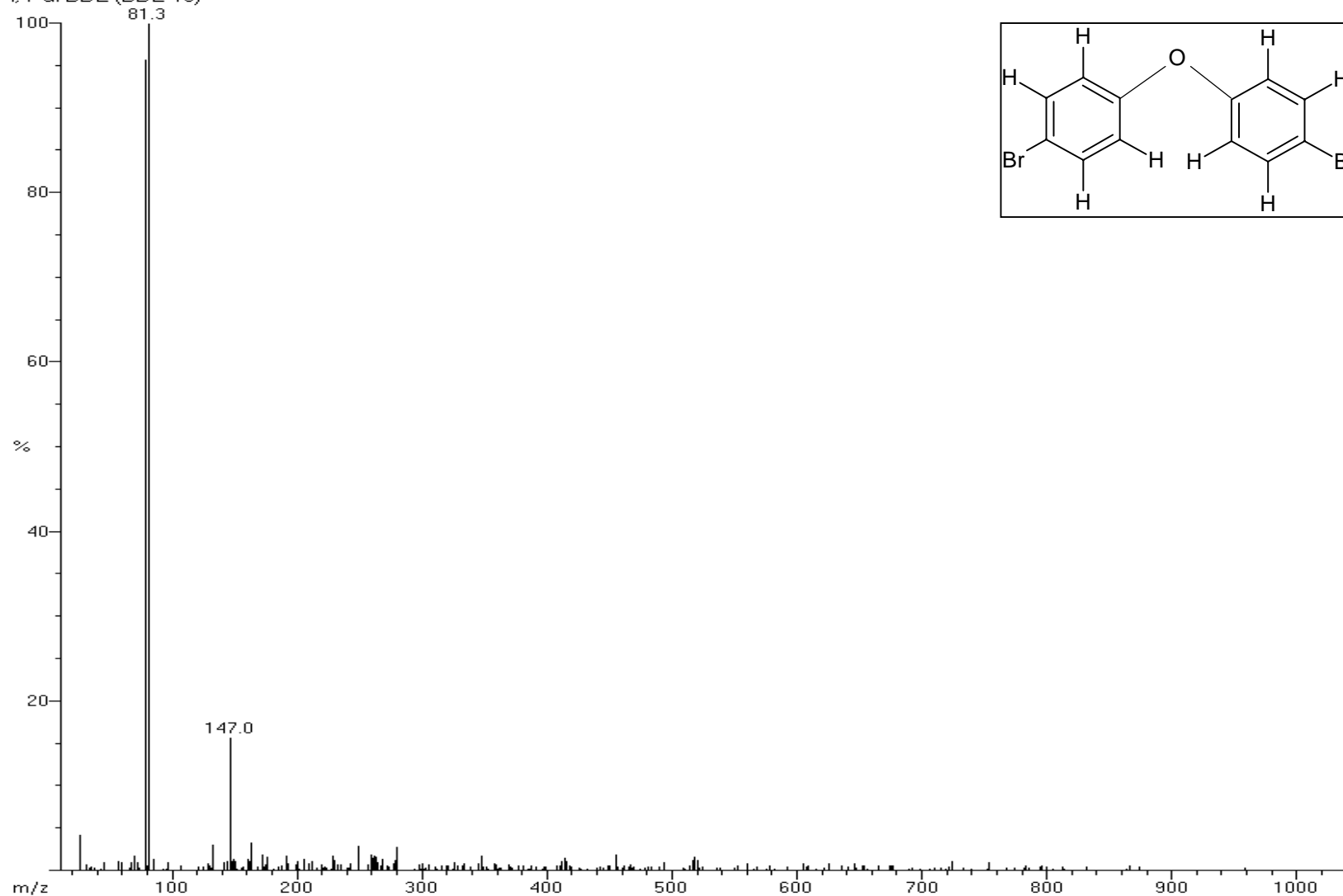
2,4-di BDE (BDE-7)



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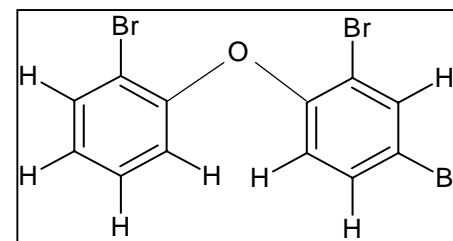
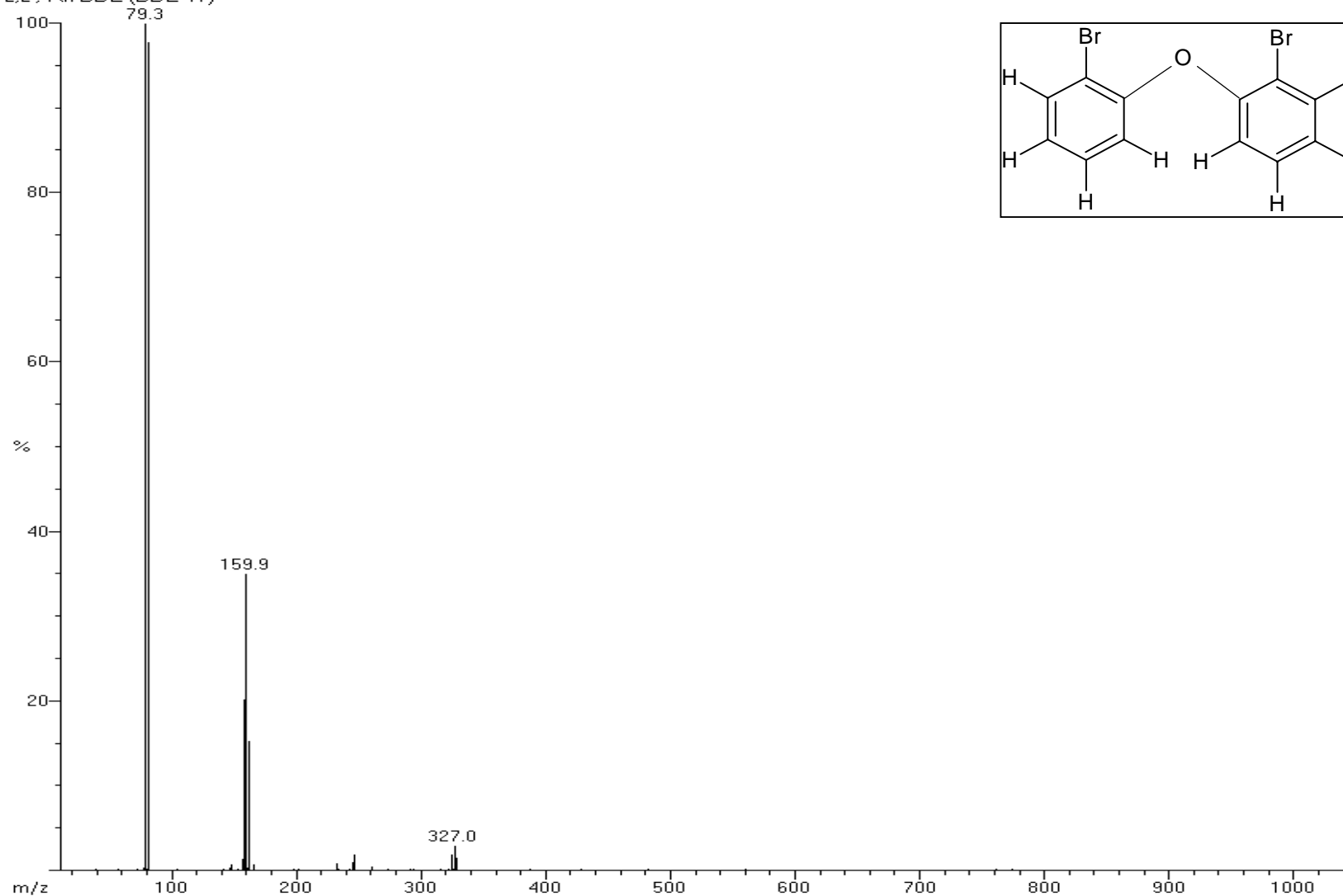
4,4'-di BDE (BDE-15)



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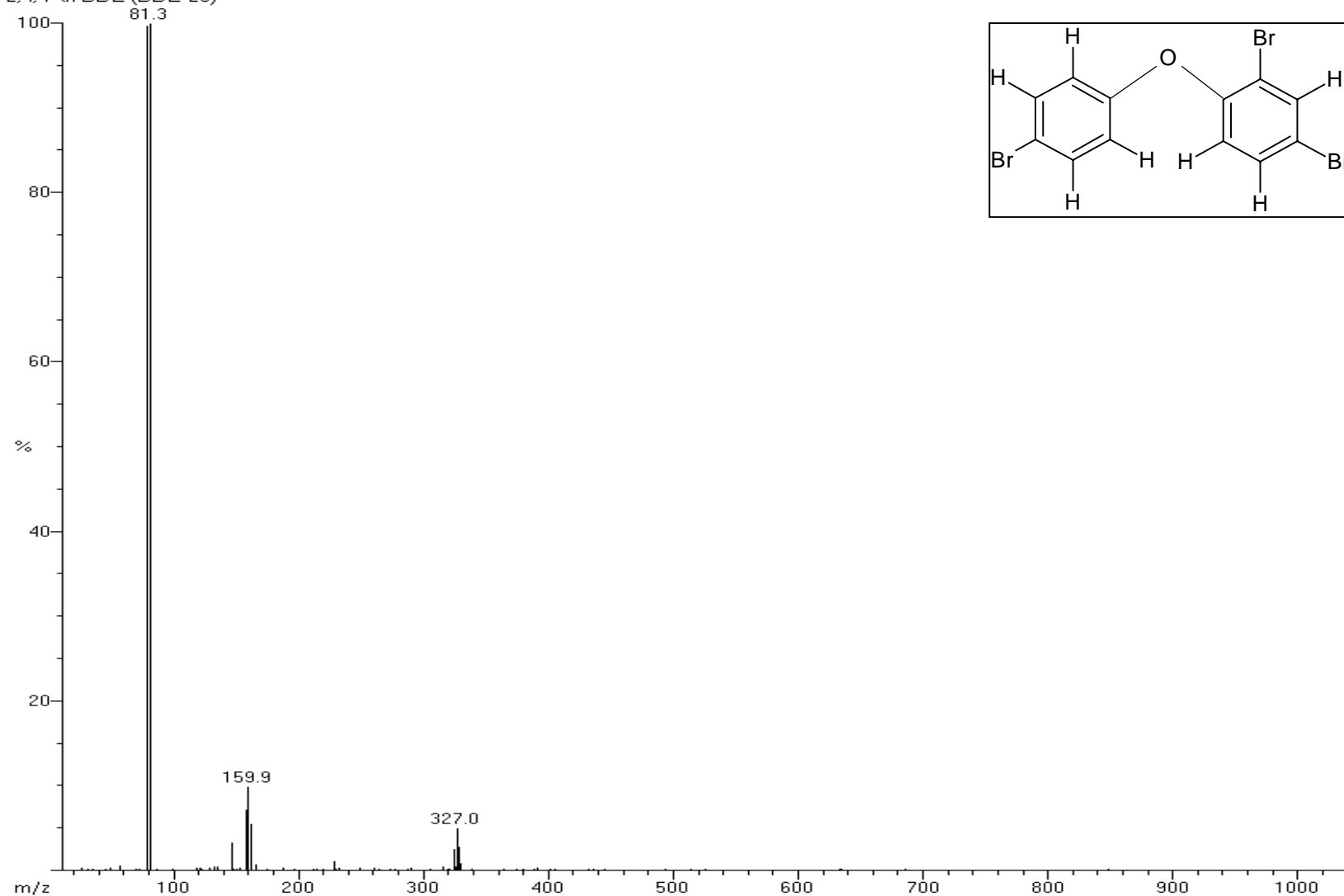
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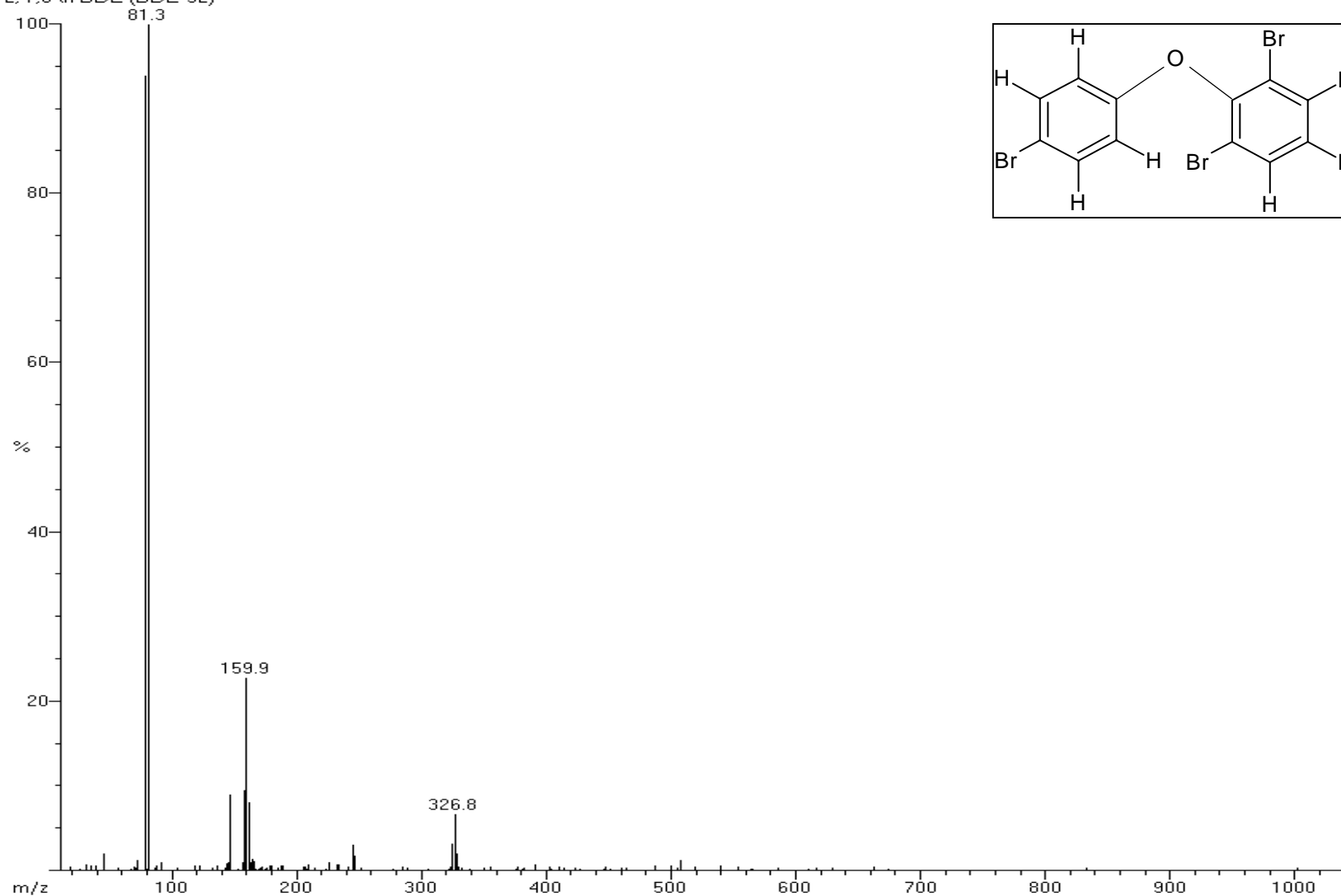
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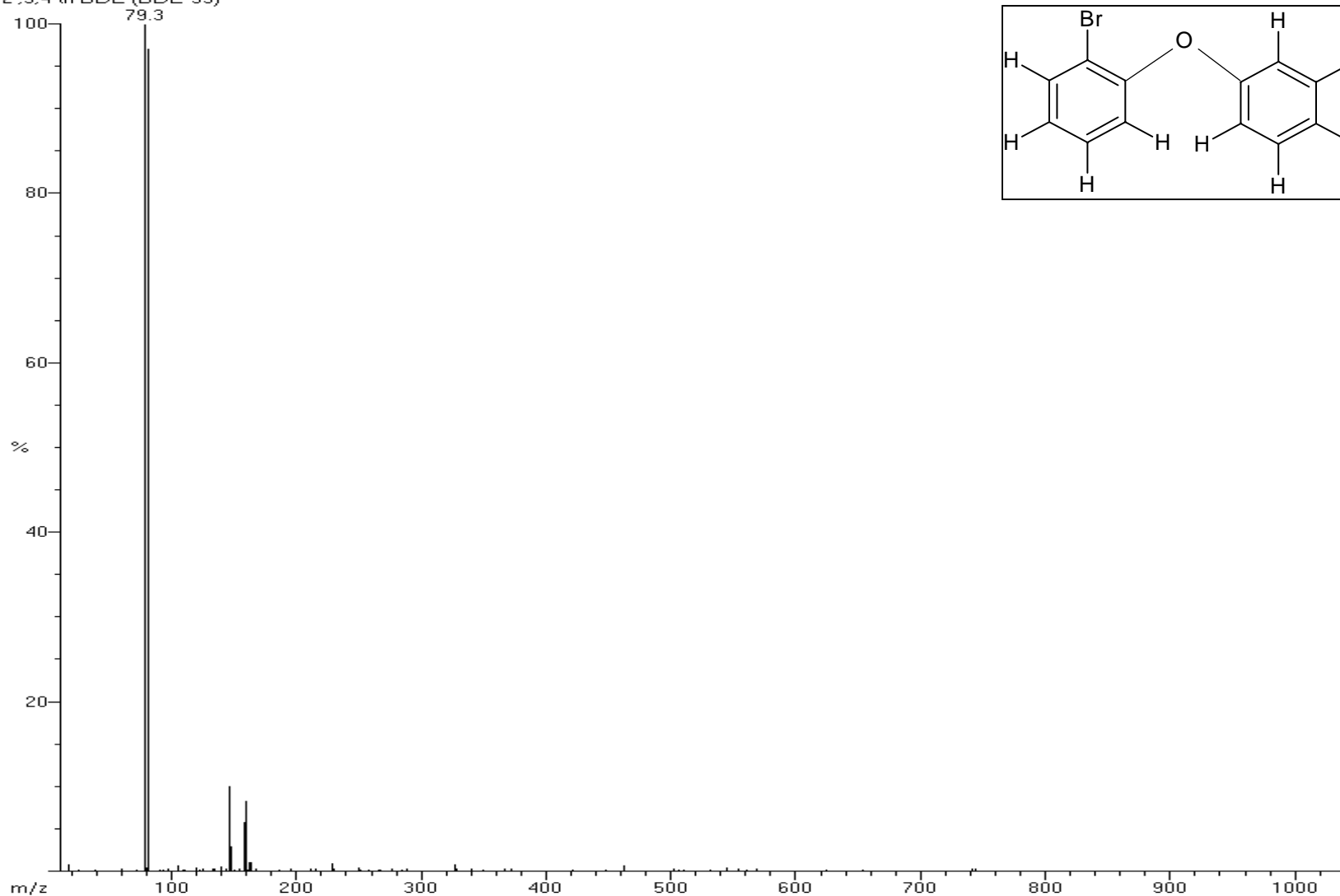
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2,4,6-tri BDE (BDE-32)



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bde33 | bde33 450pg  
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2,3,4-tri BDE (BDE-33)

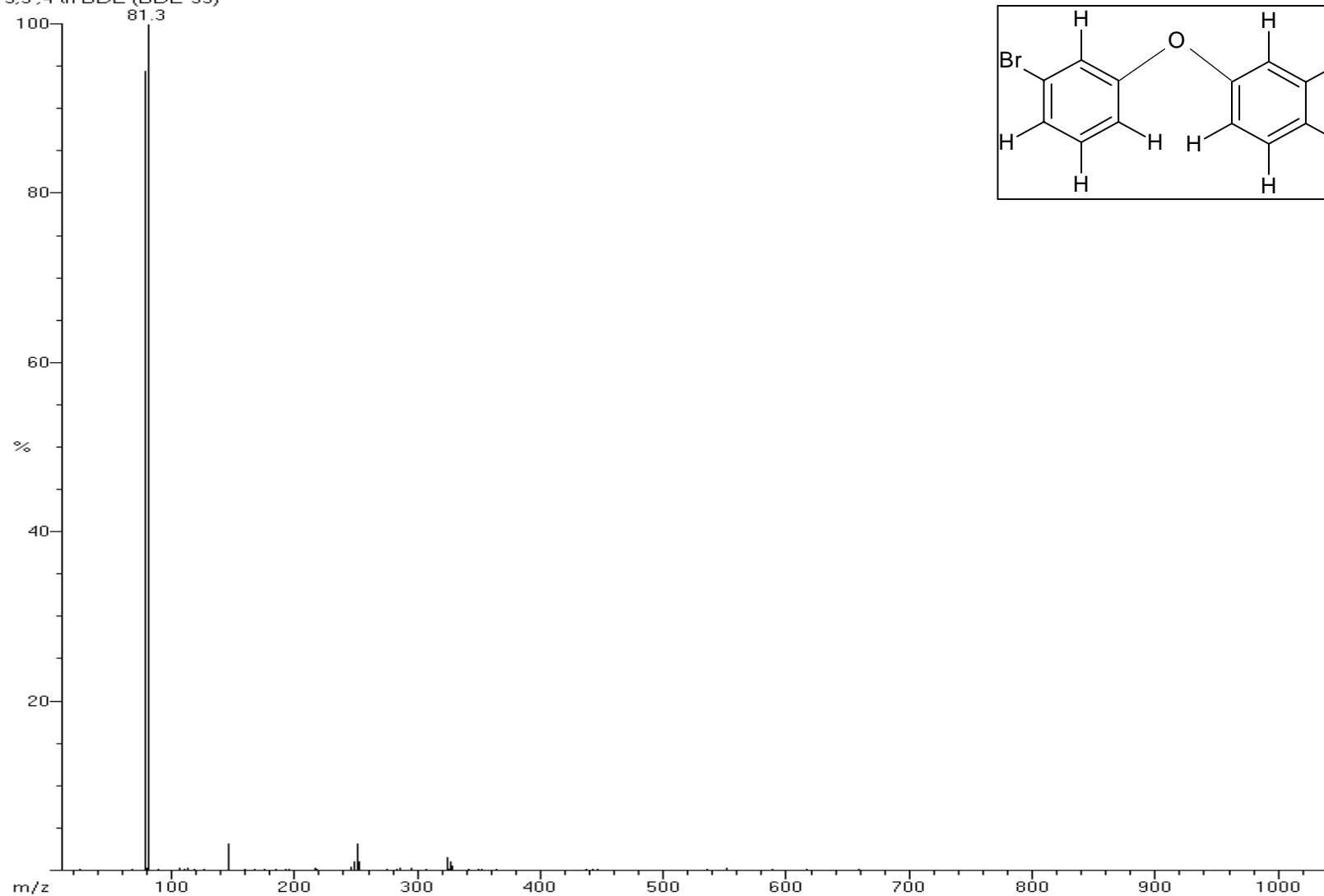


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bde35 | bde35 450pg  
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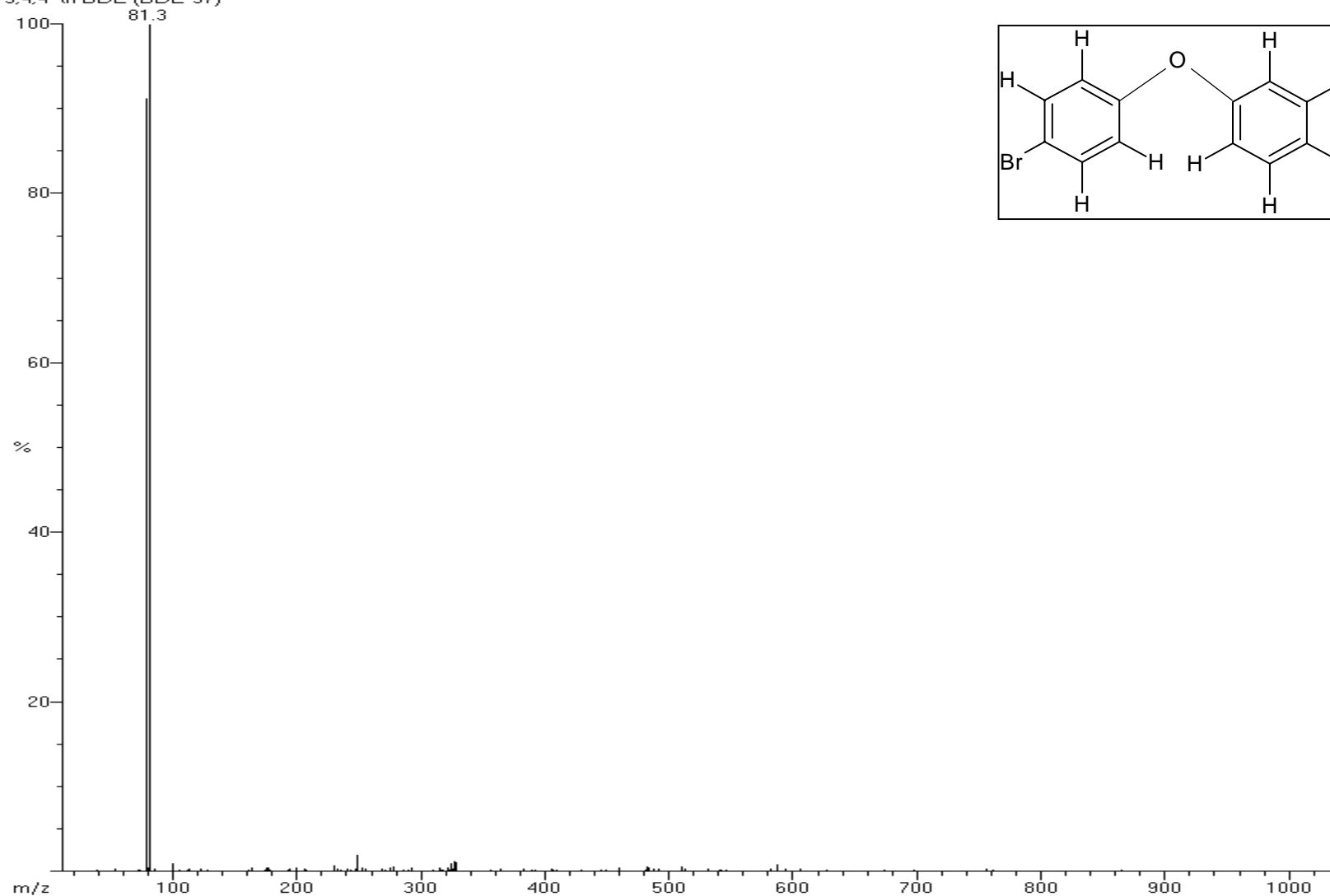
3,3',4-tri BDE (BDE-35)



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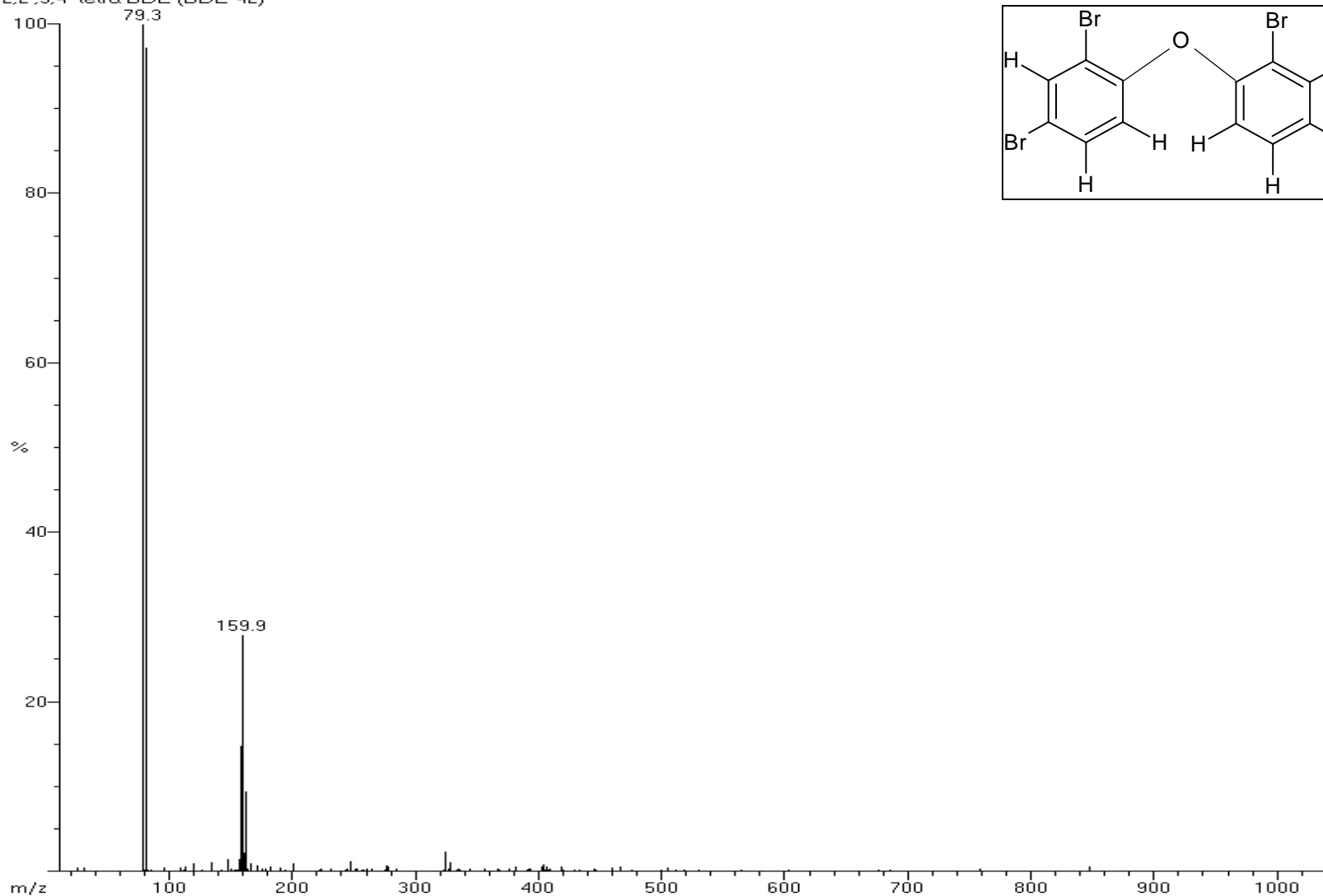
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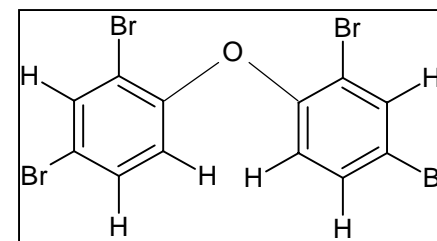
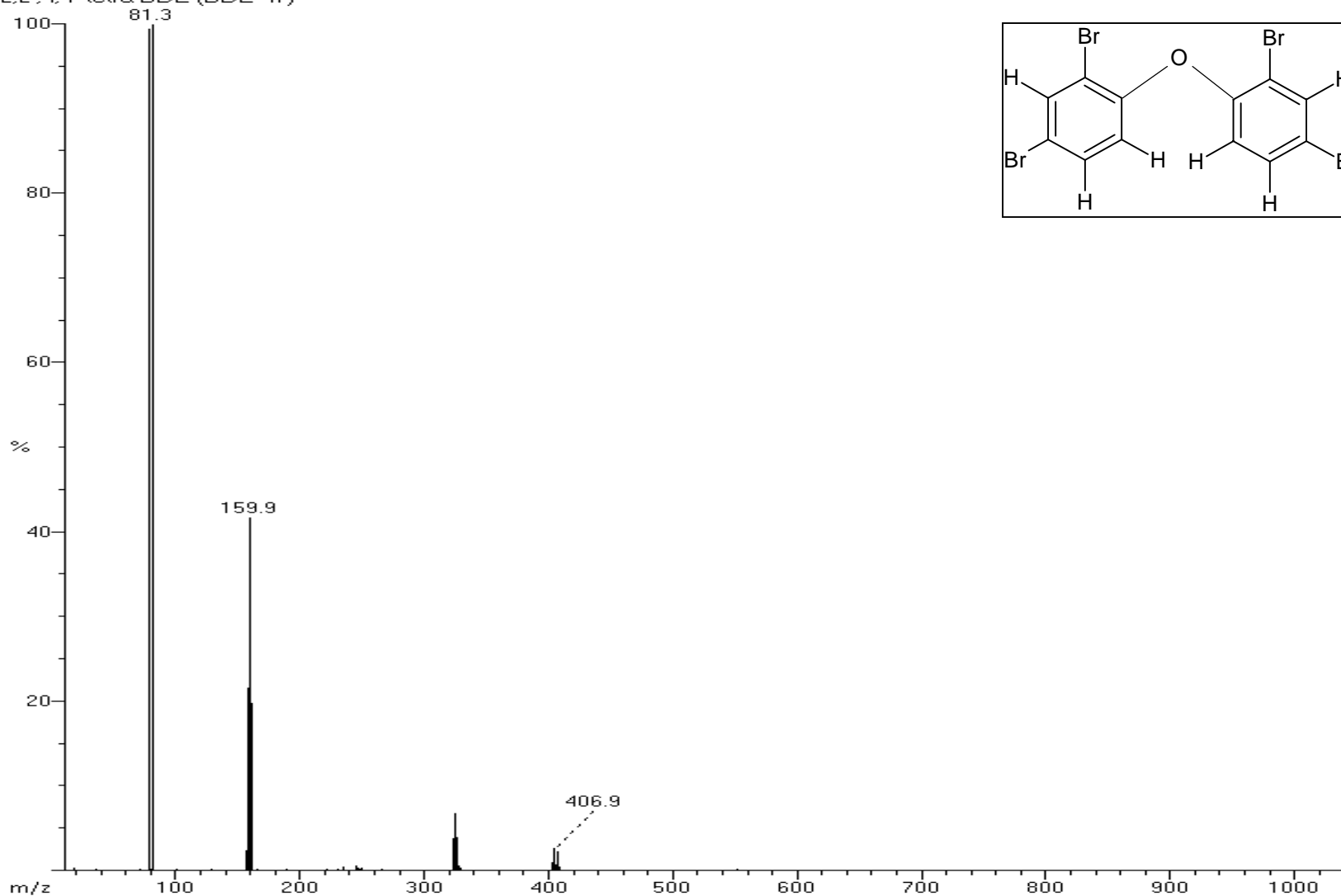
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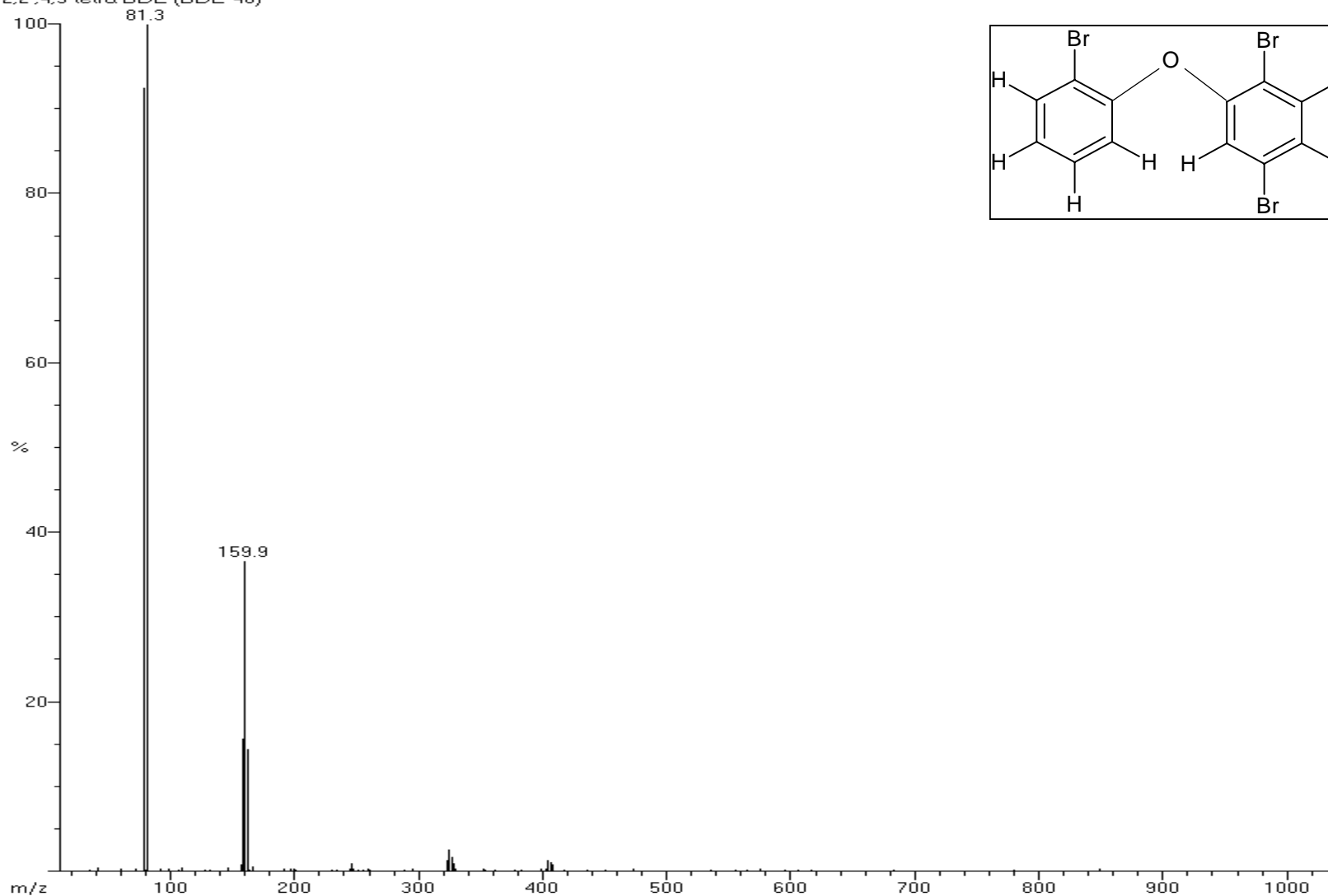
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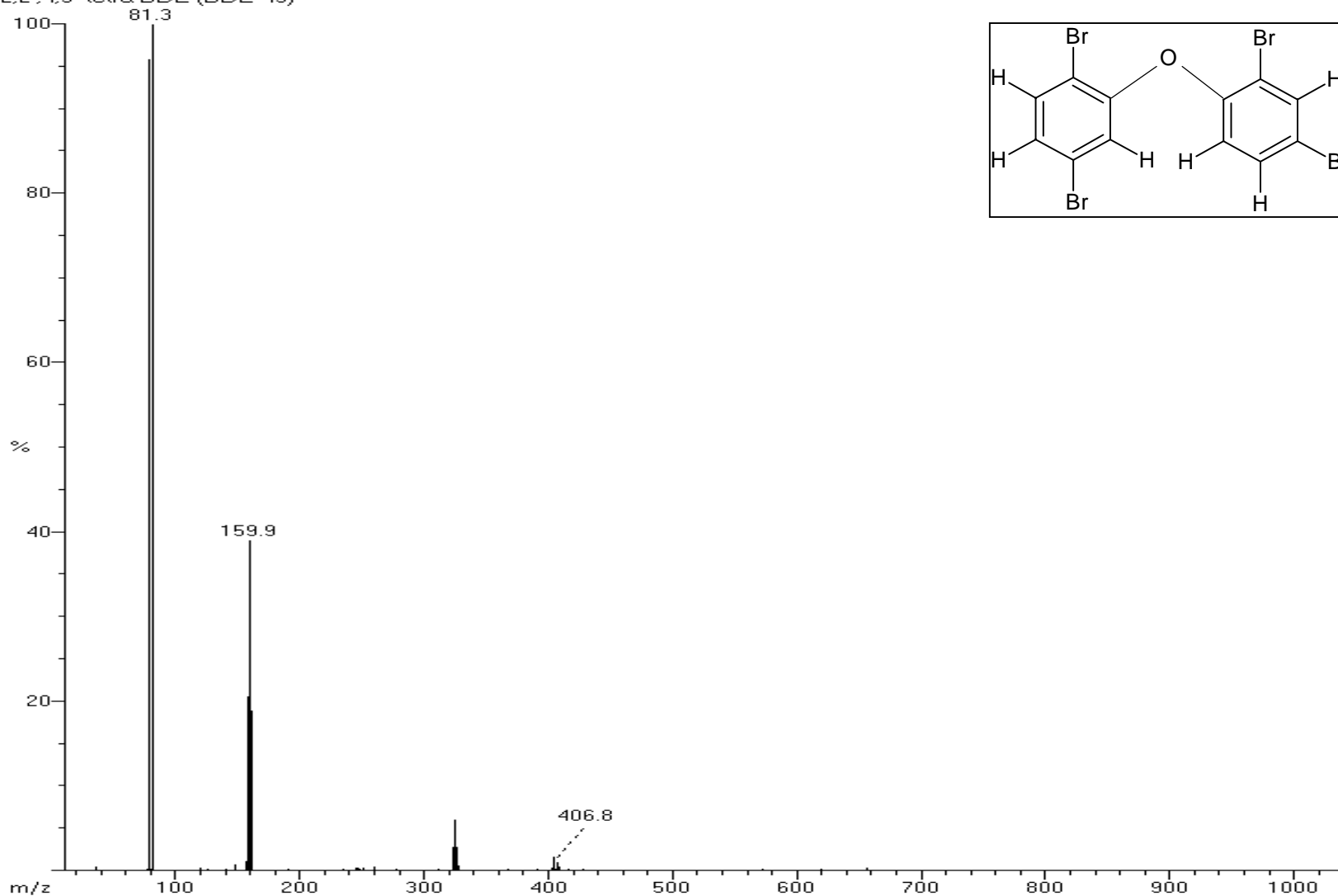
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2,2',4,5-tetra BDE (BDE-48)



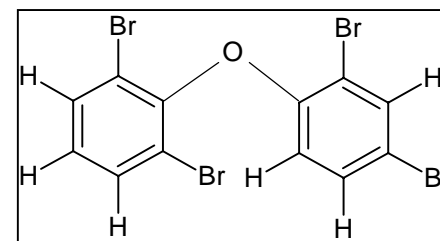
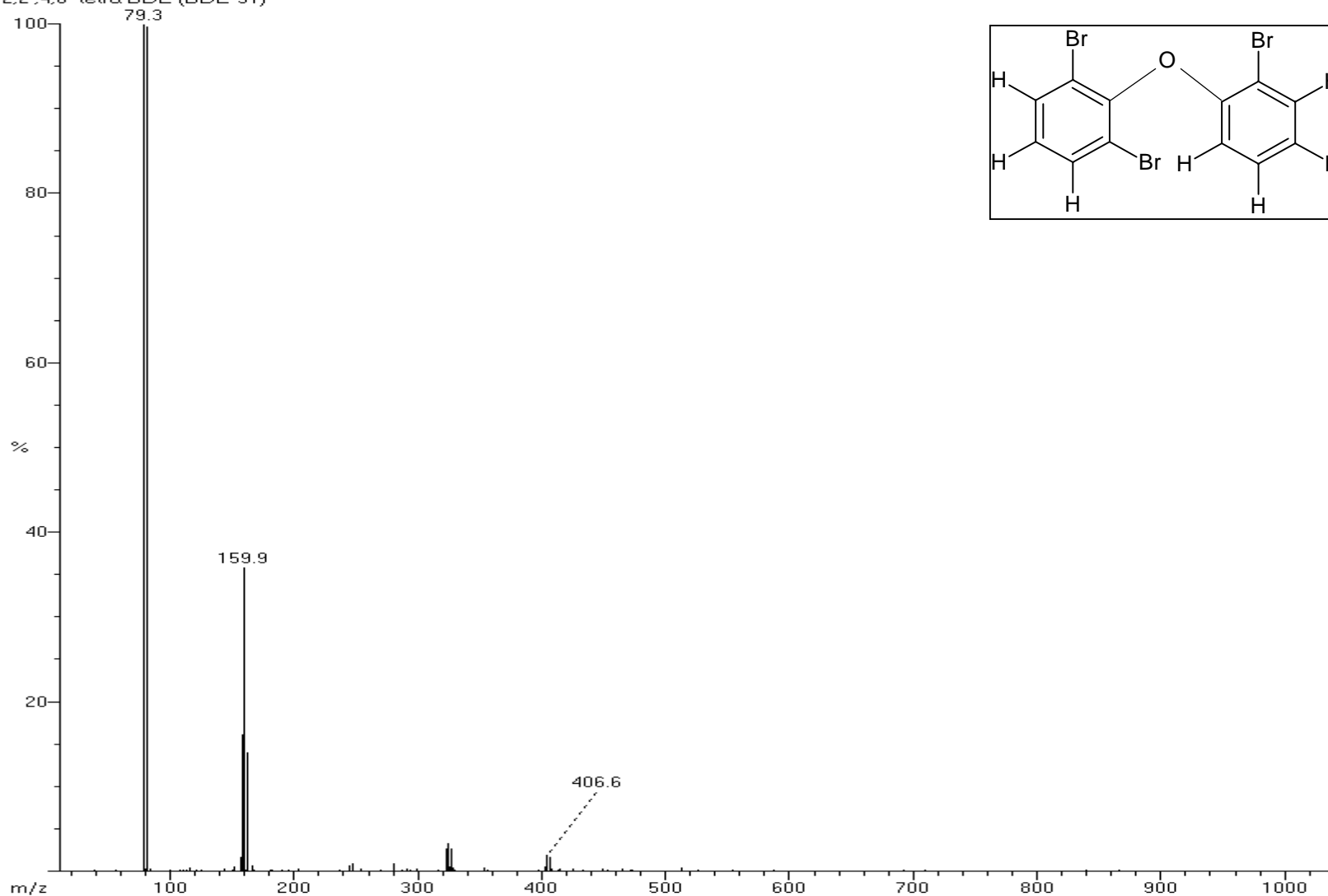
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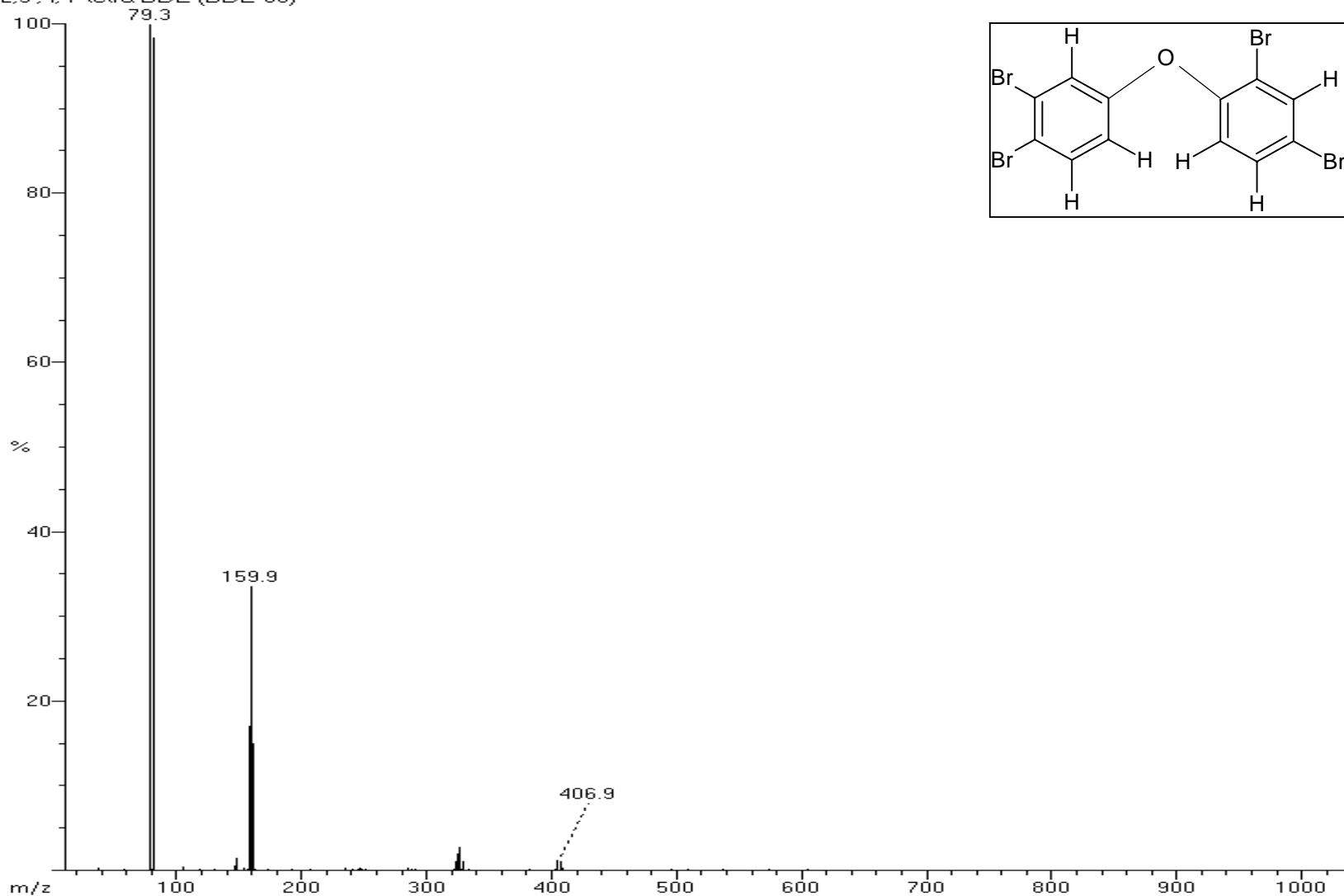


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bde51 | bde51 1000pg  
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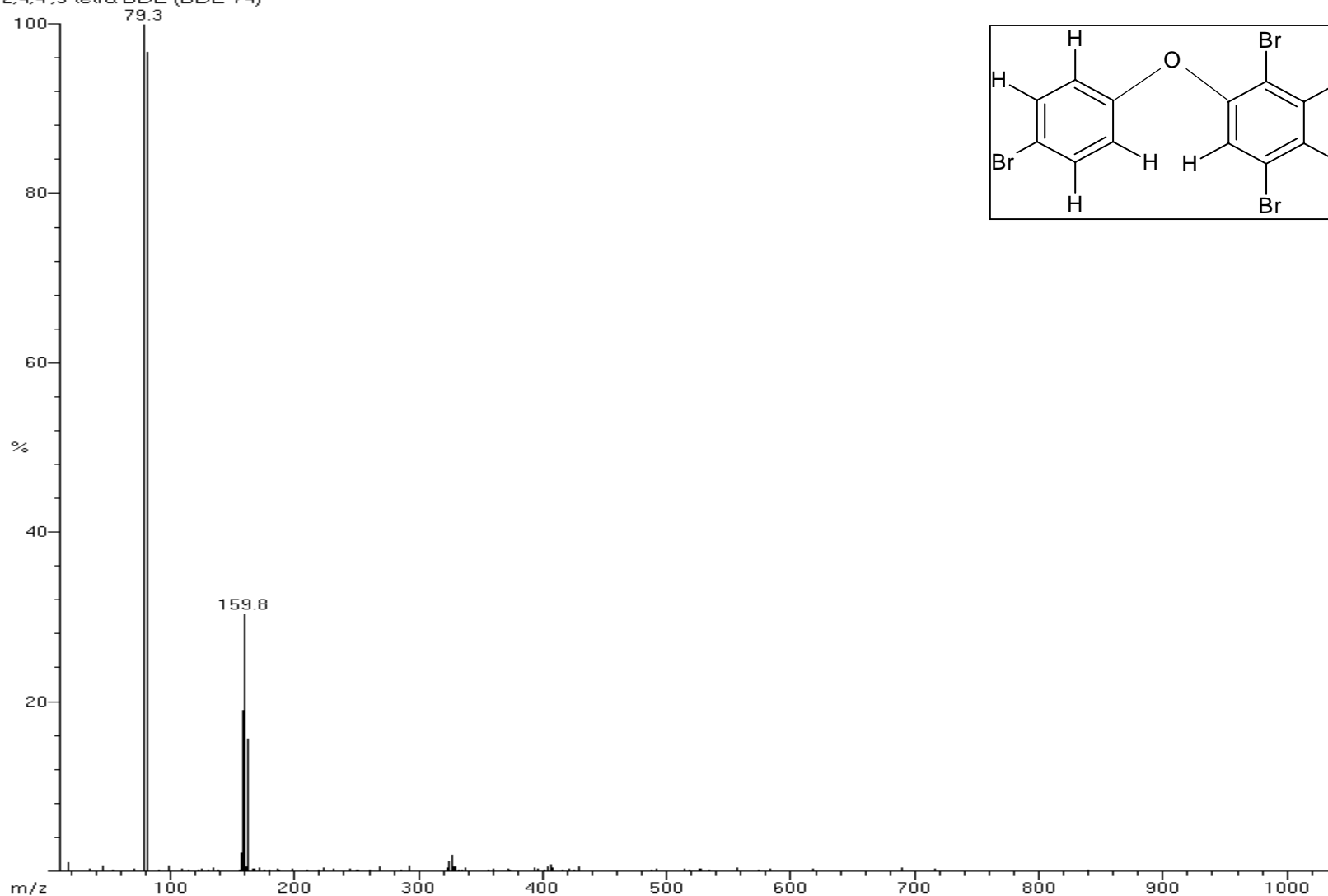
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2,3',4,4'-tetra BDE (BDE-66)



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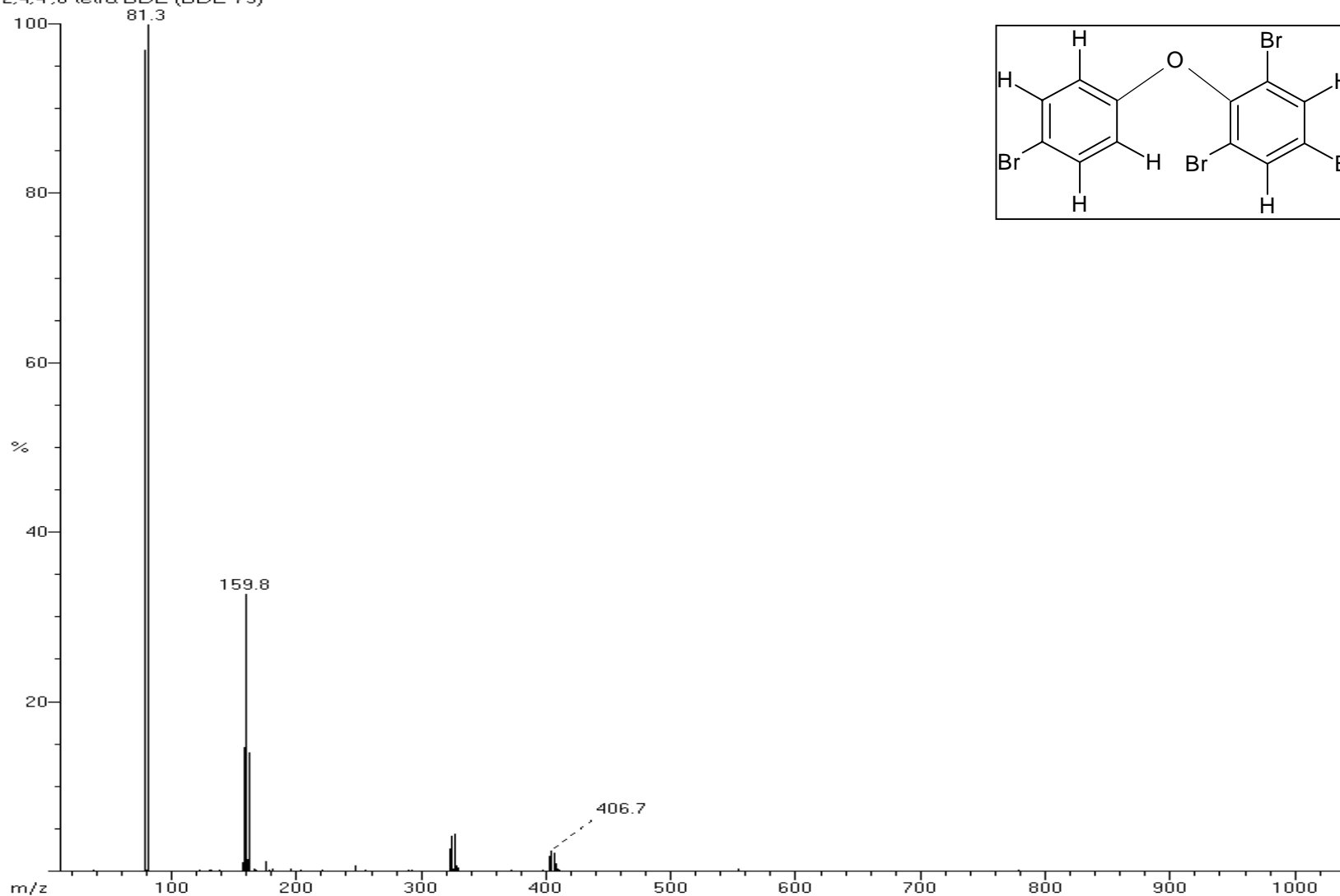
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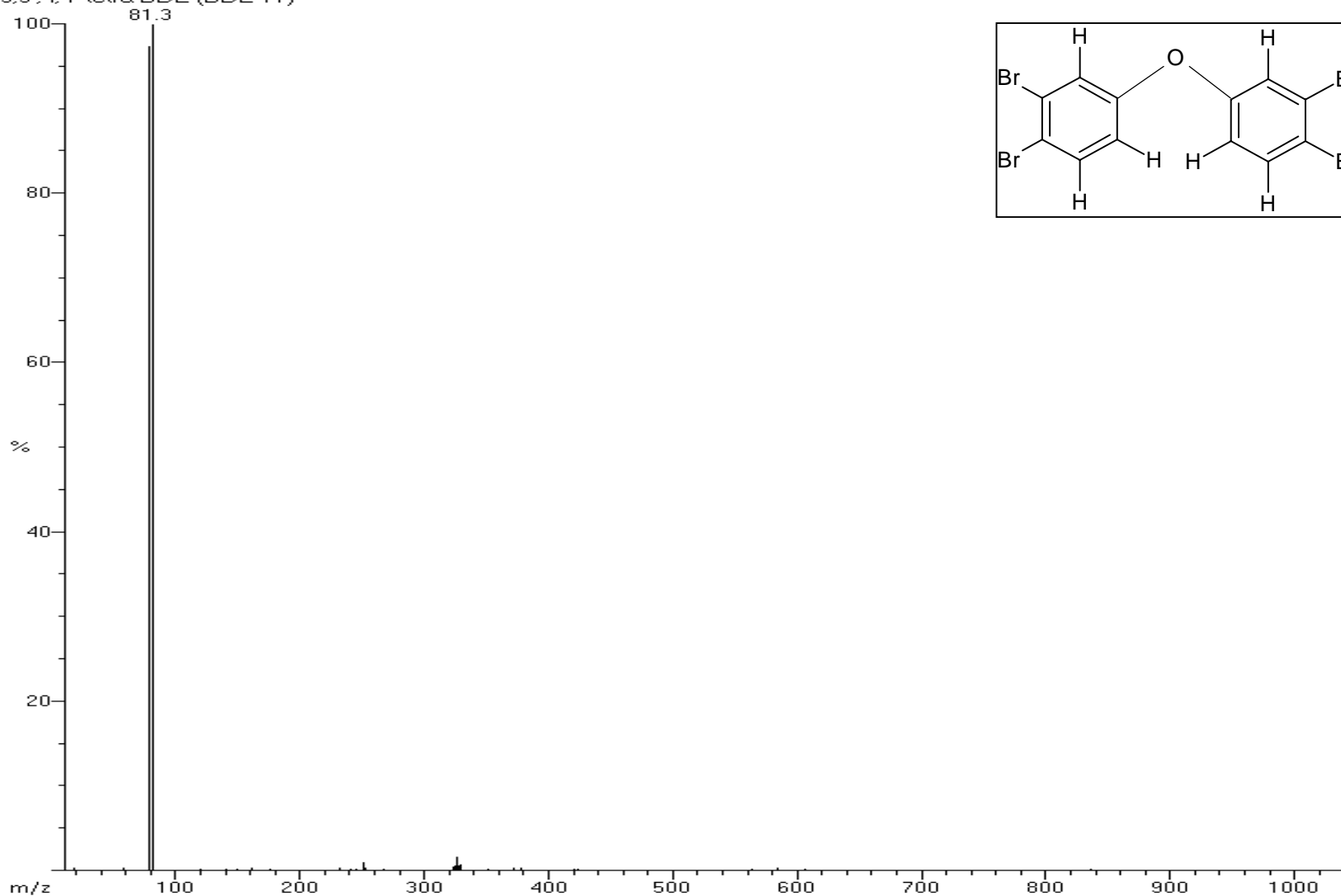
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2,4,4',6-tetra BDE (BDE-75)



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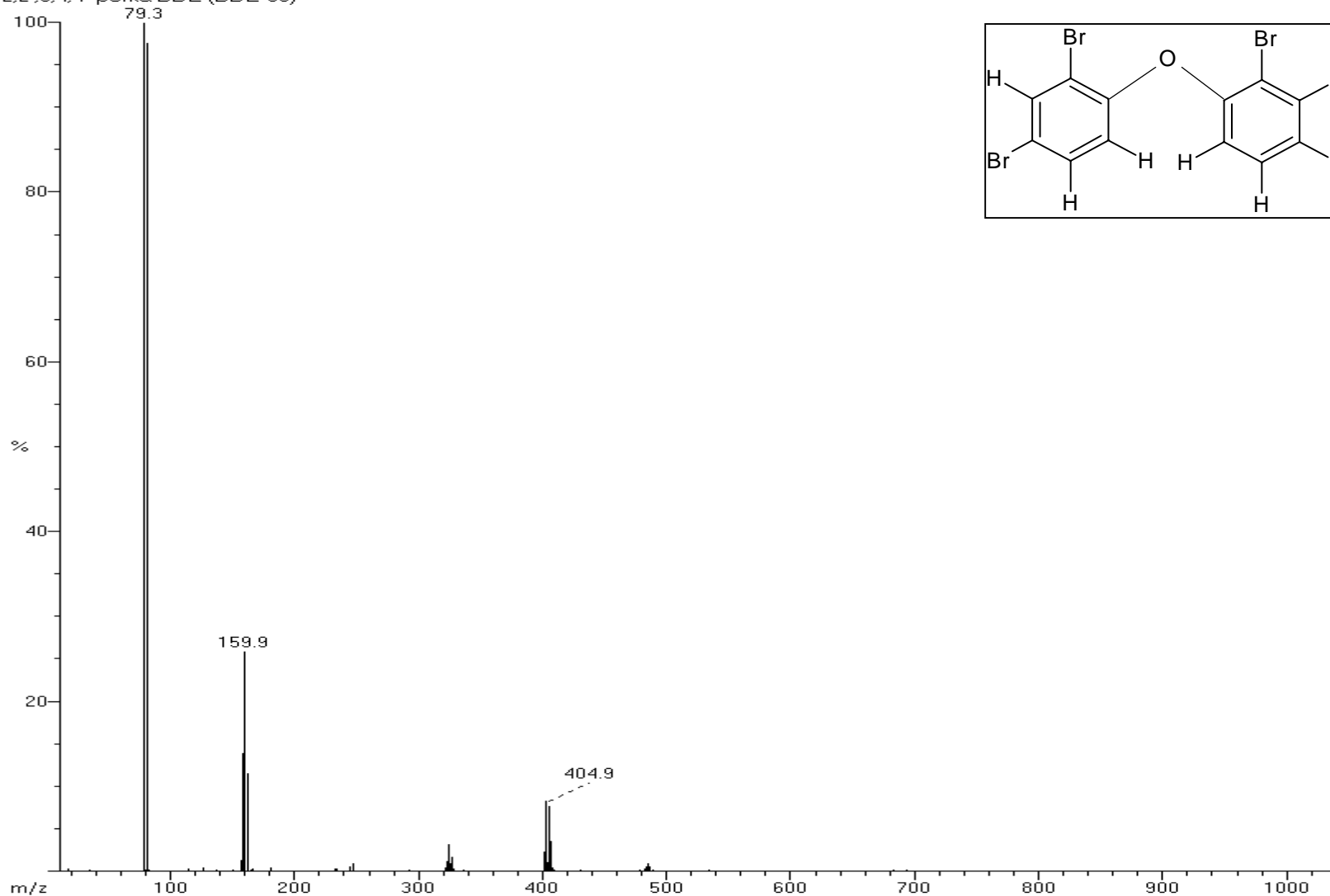
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3,3',4,4'-tetra BDE (BDE-77)



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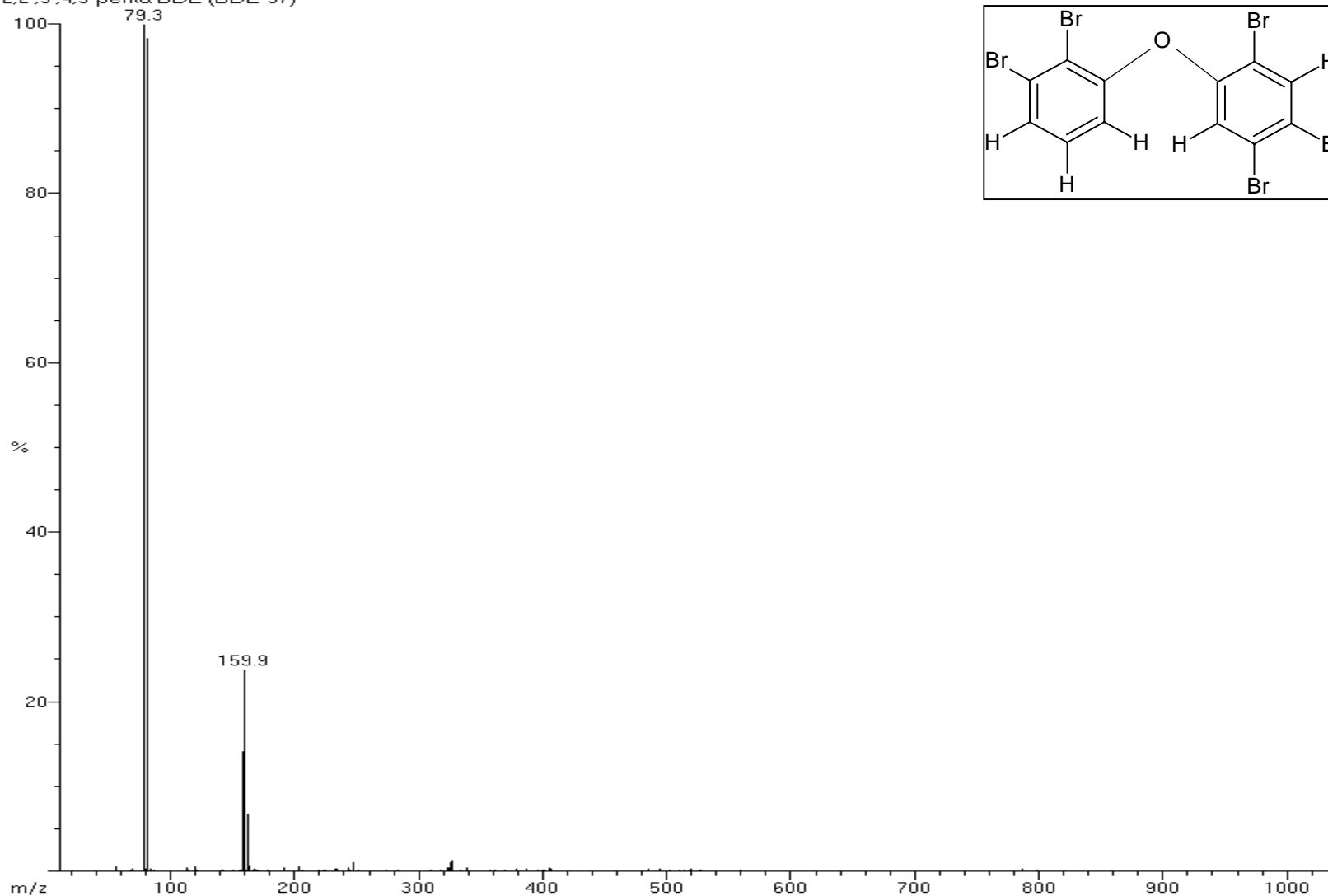
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2,2',3,4,4'-penta BDE (BDE-85)



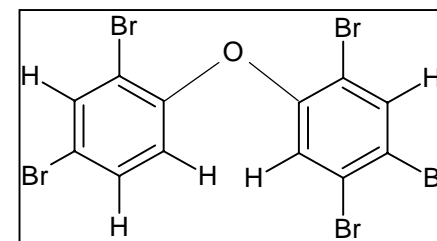
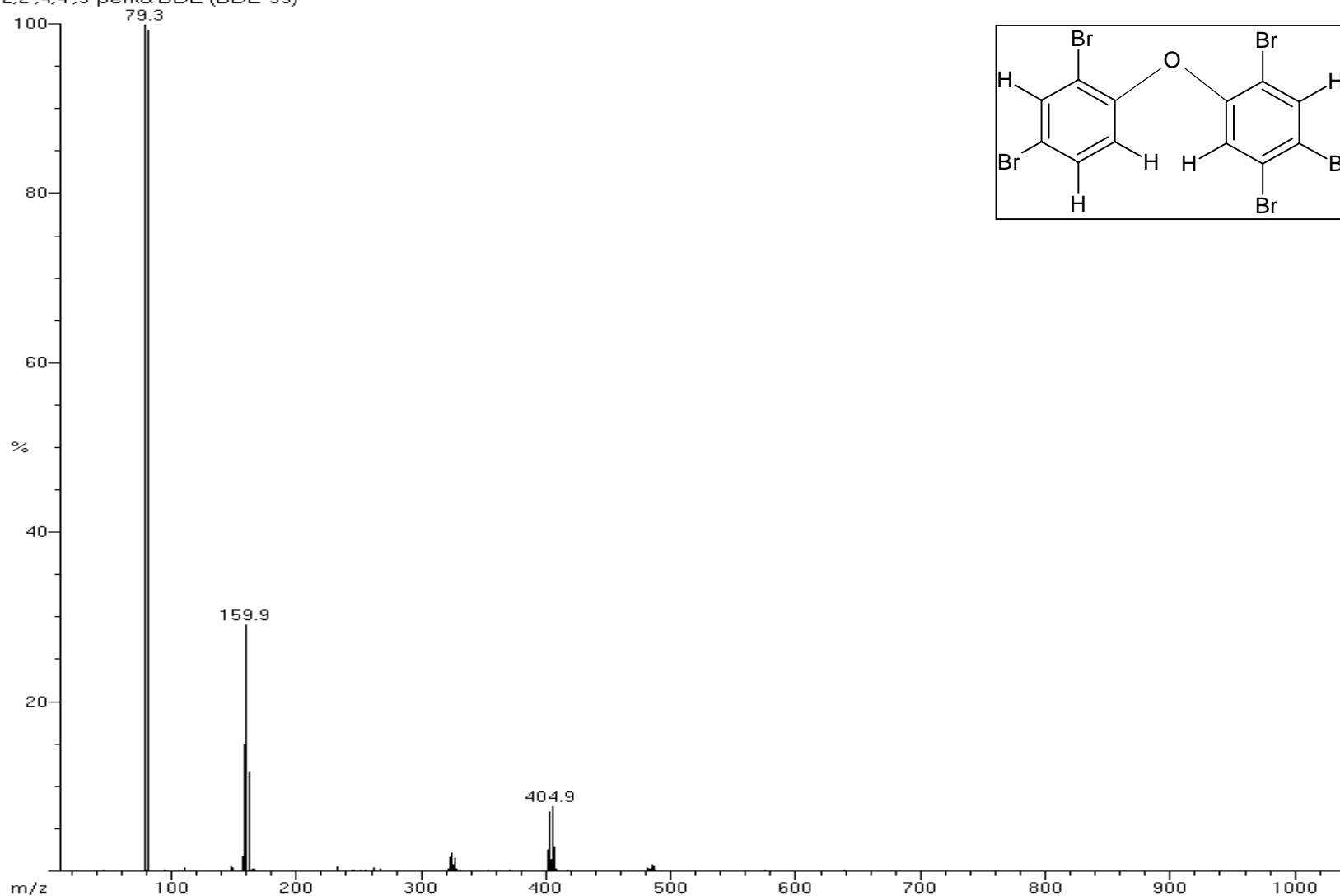
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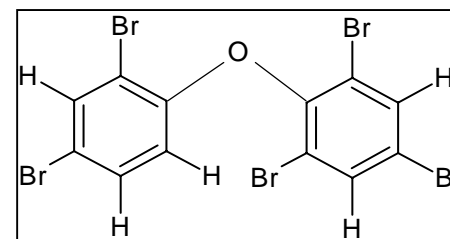
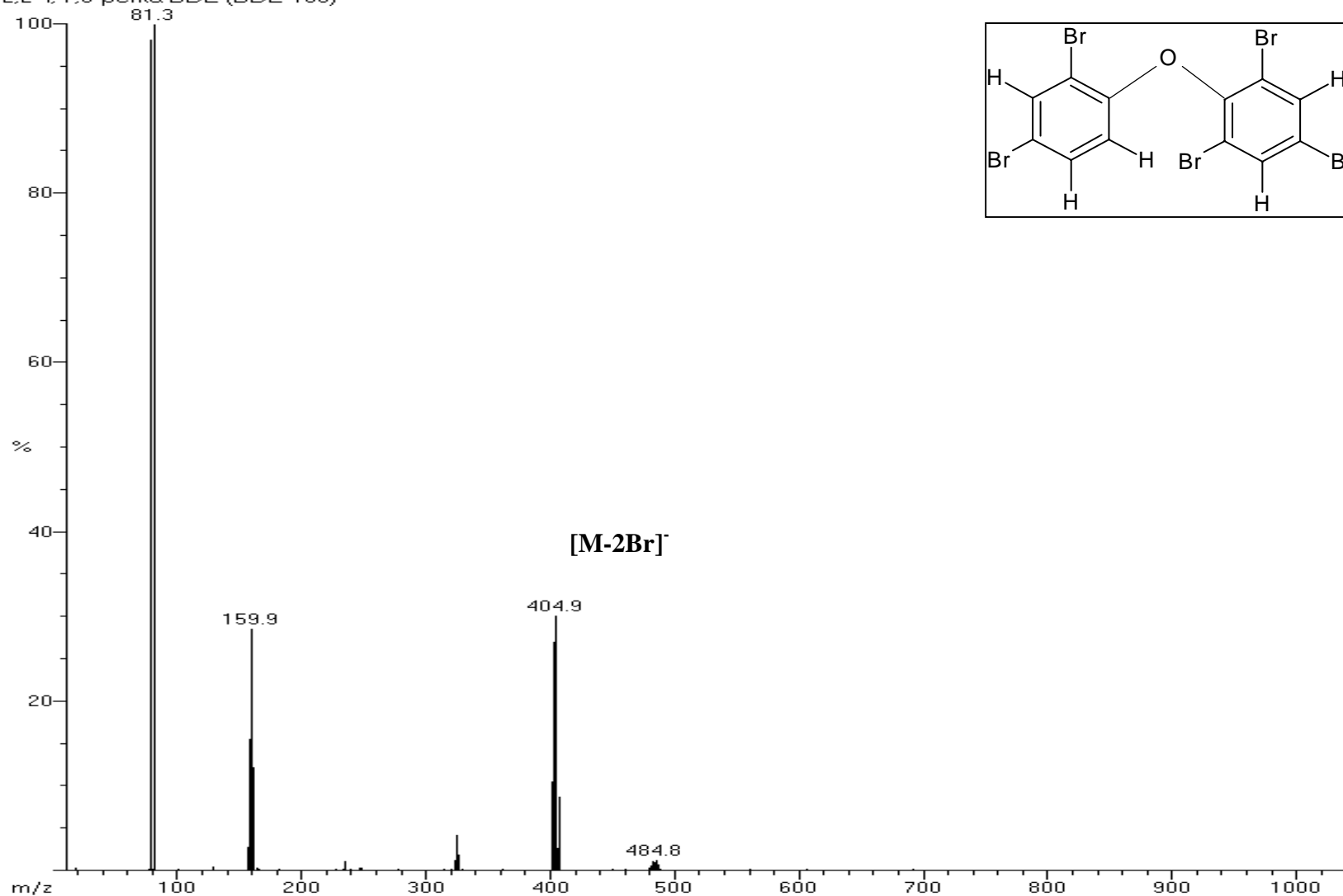
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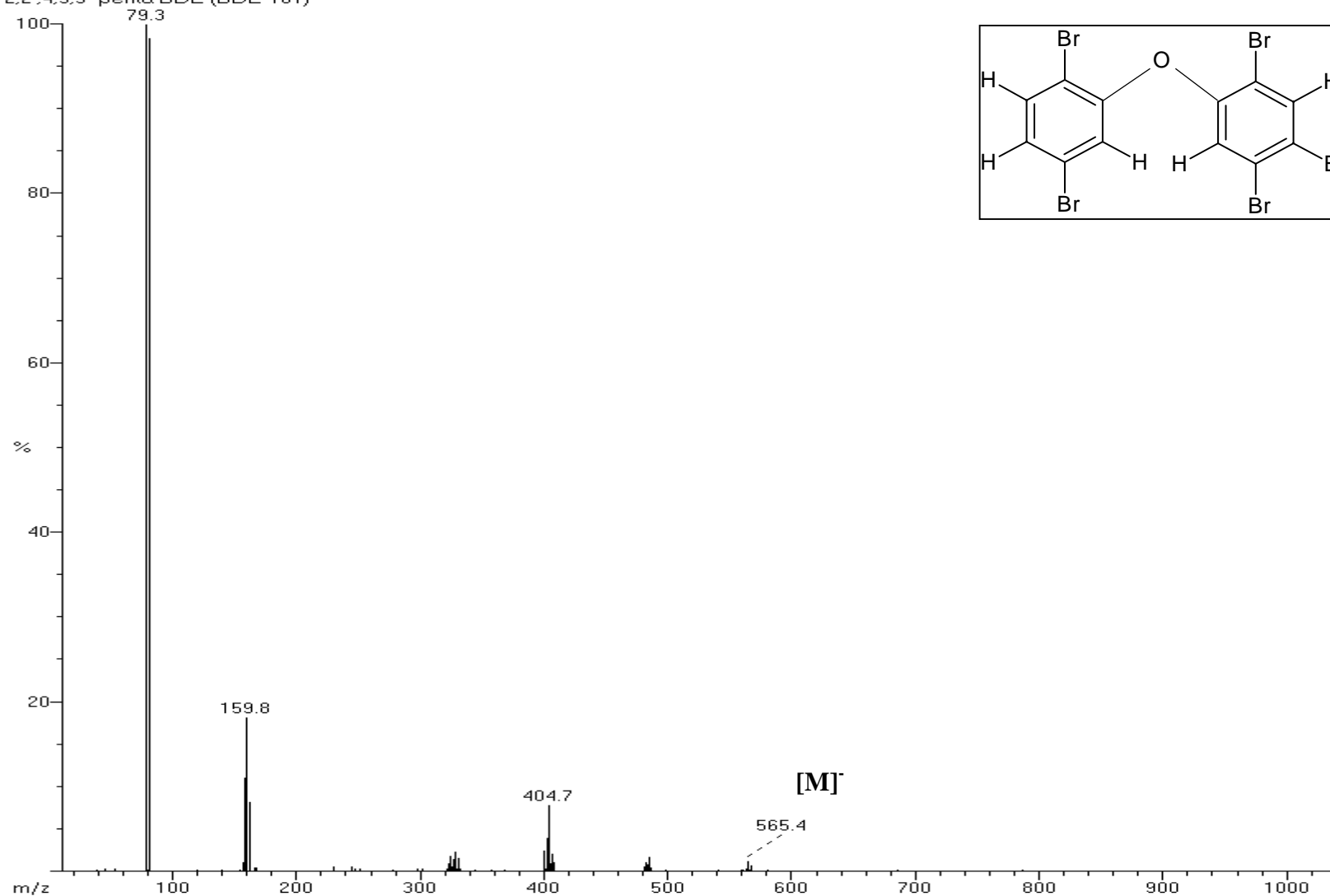
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2,2',4,4',6-penta BDE (BDE-100)



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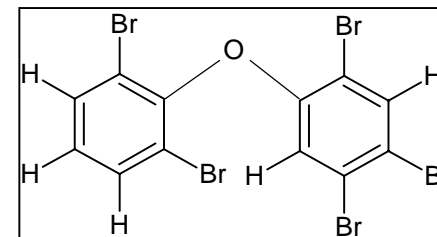
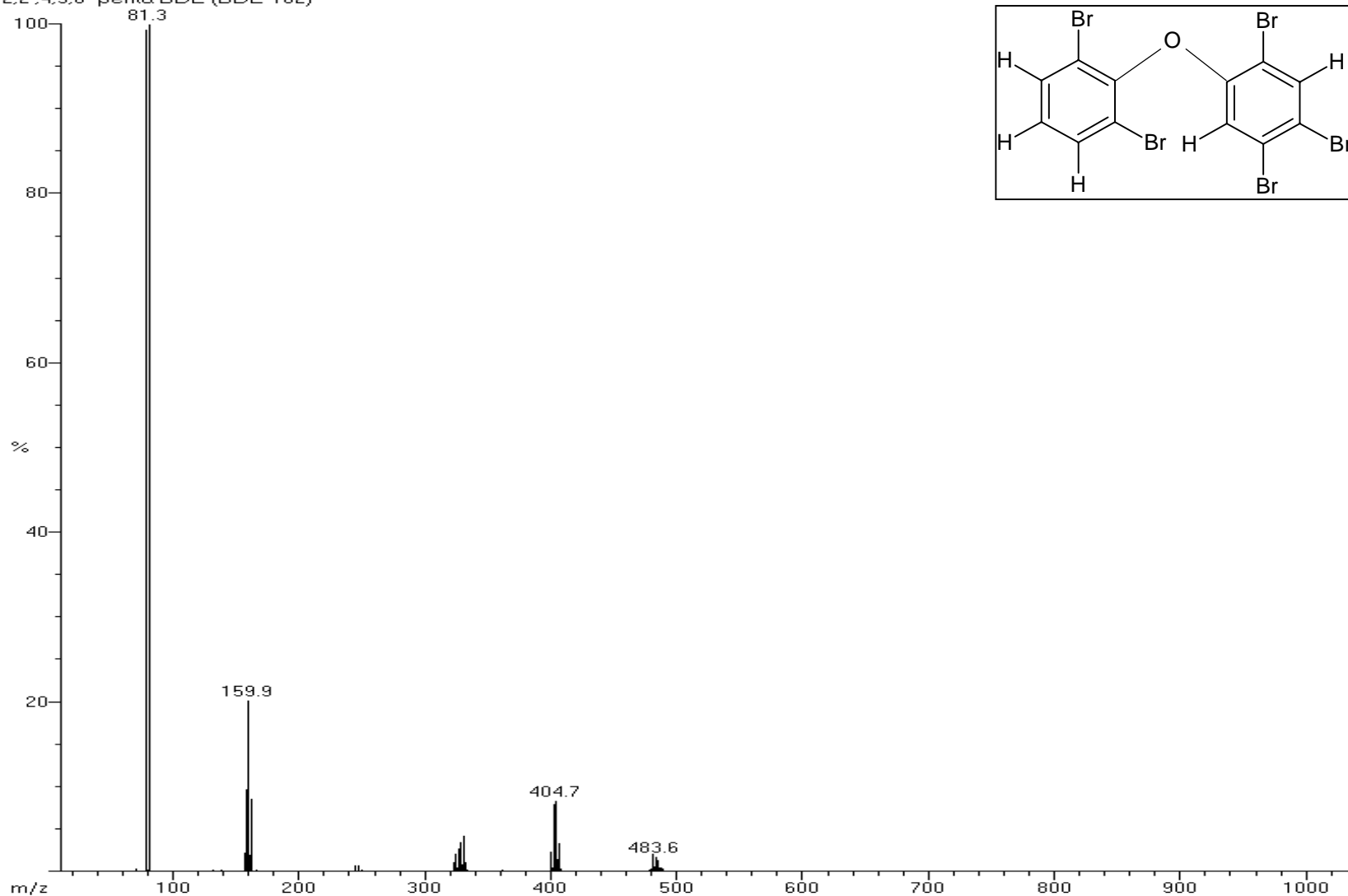
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2,2',4,5,5'-penta BDE (BDE-101)



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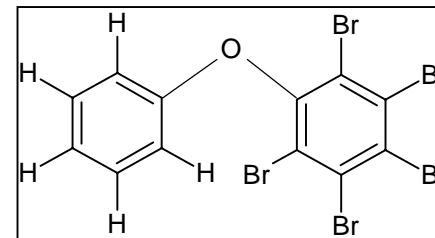
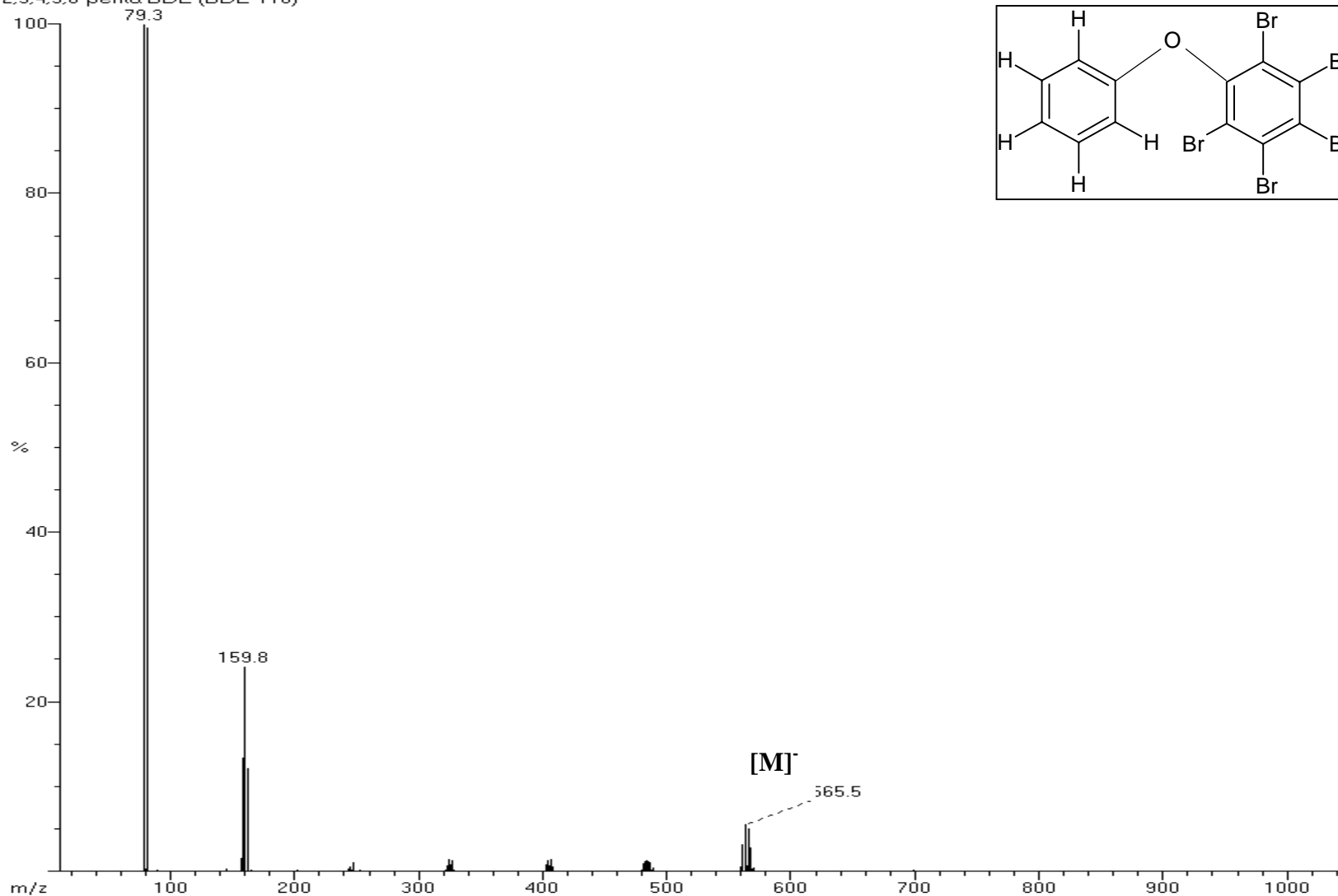


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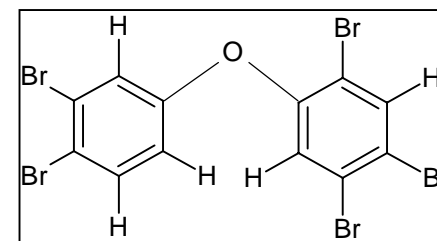
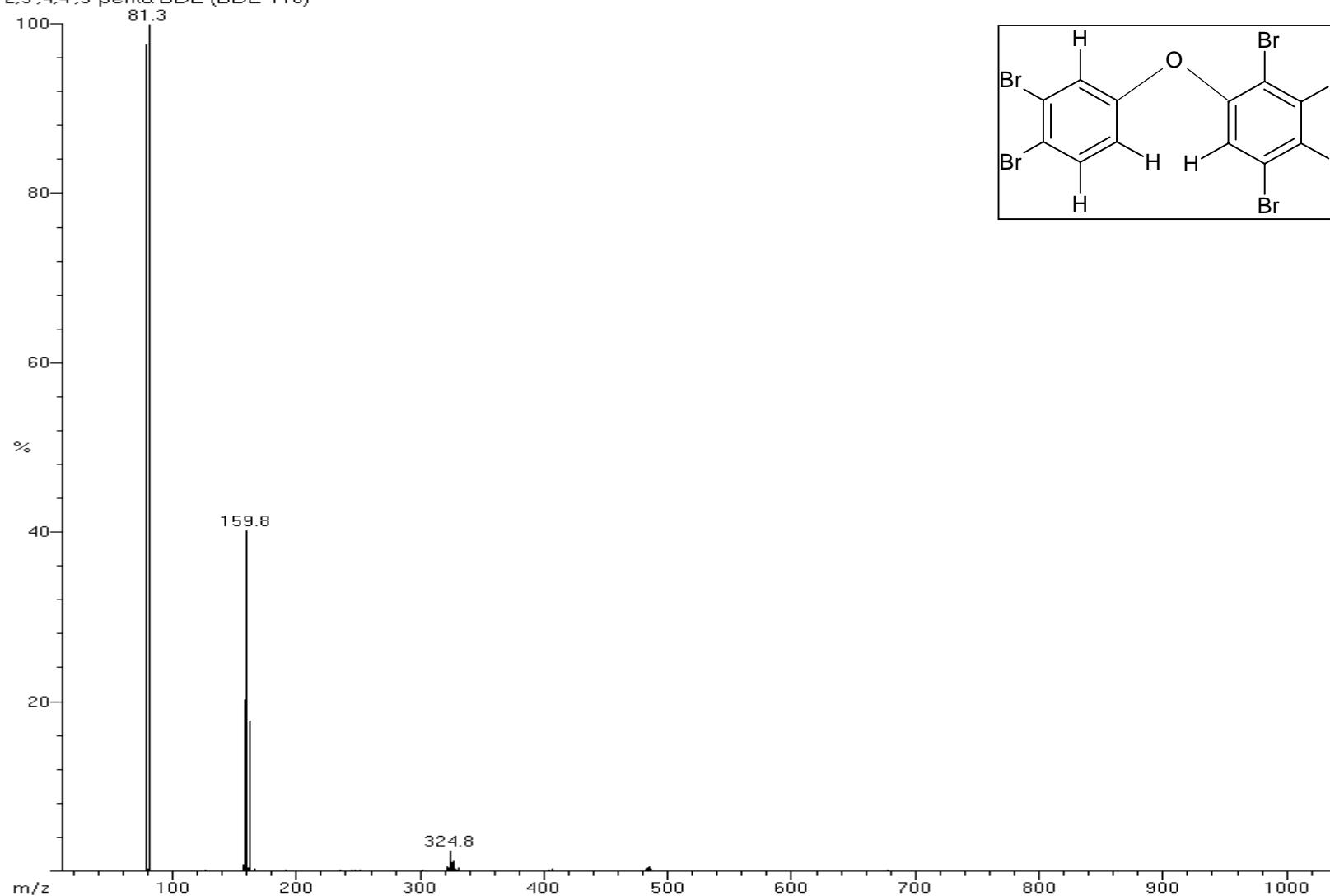
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bde116 | bde116 450pg  
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2,3,4,5,6-penta BDE (BDE-116)



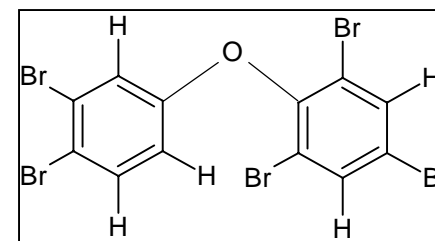
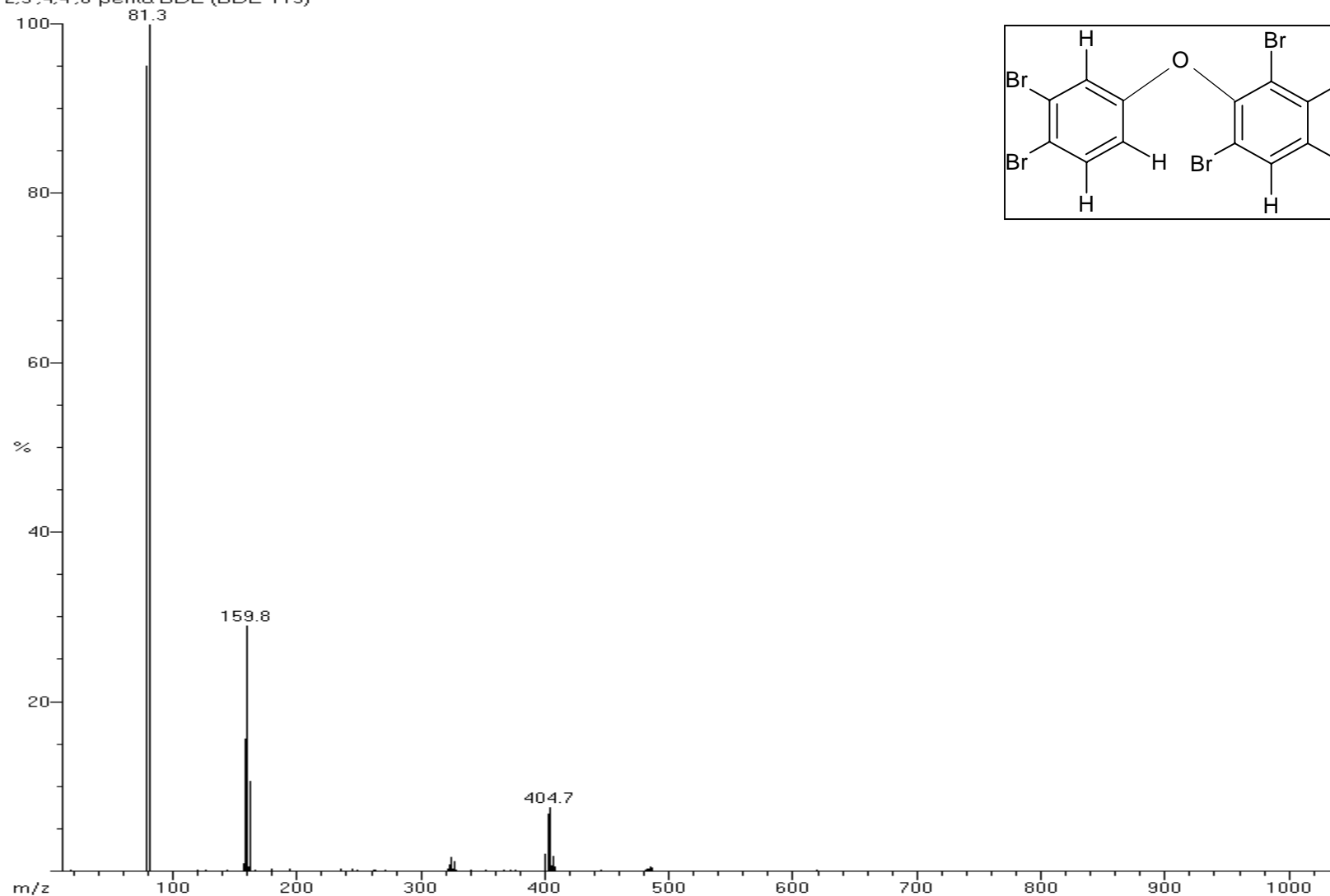
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bde118 | bde118 5000pg  
Scan: 457(428-442) TIC=2508777 Base=76.7%FS #ions=1048 RT=14.3  
2,3',4,4',5-penta BDE (BDE-118)



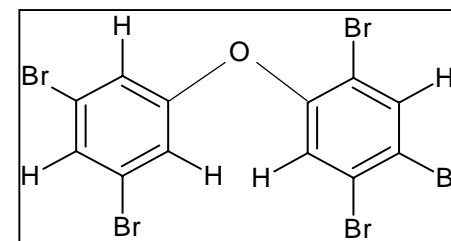
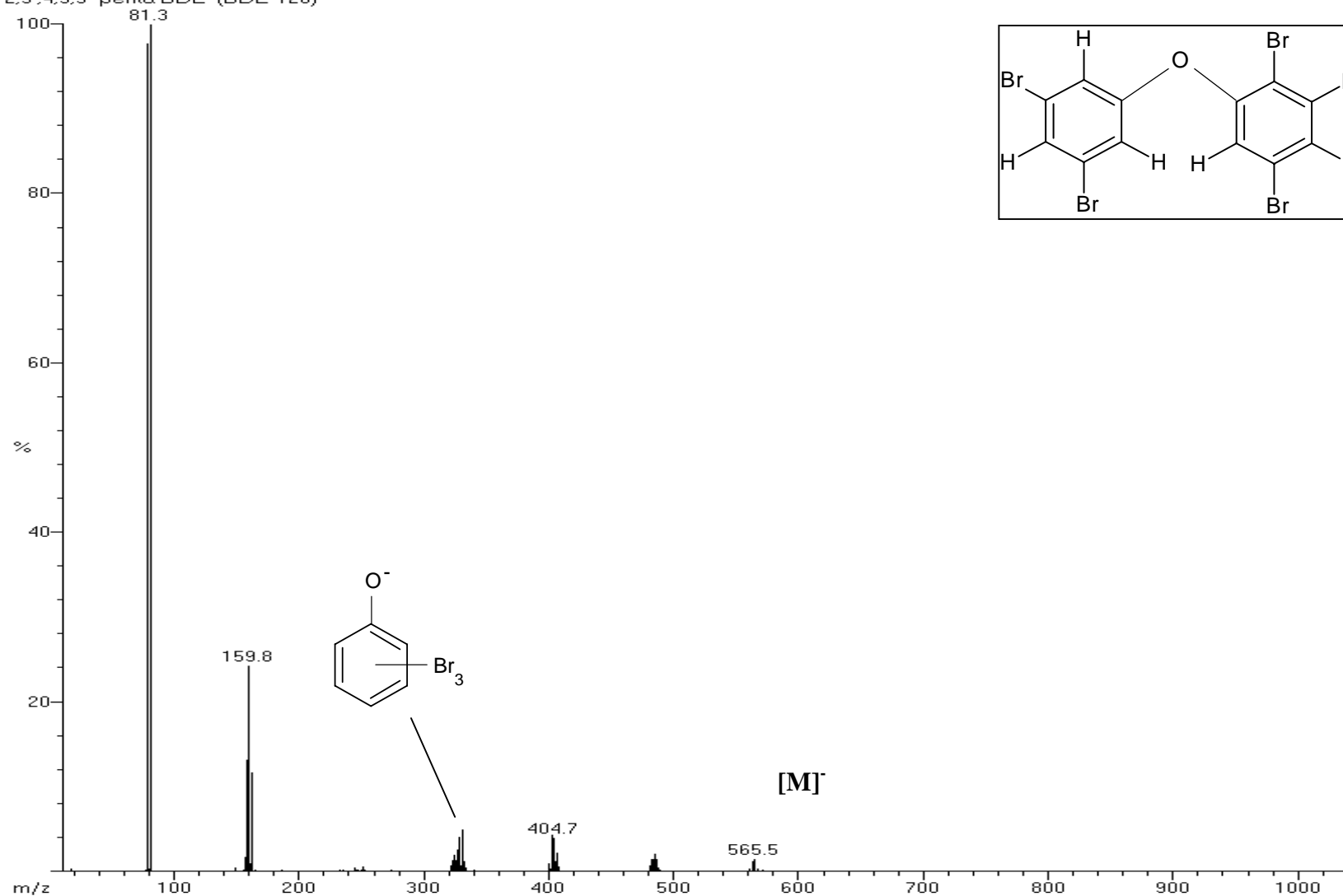
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*Gloucester Point, Virginia 23062*

bde119 | bde119 400pg  
Scan: 426(365-379) TIC=2058159 Base=64.6%FS #ions=487 RT=13.67  
2,3',4,4',6-penta BDE (BDE-119)



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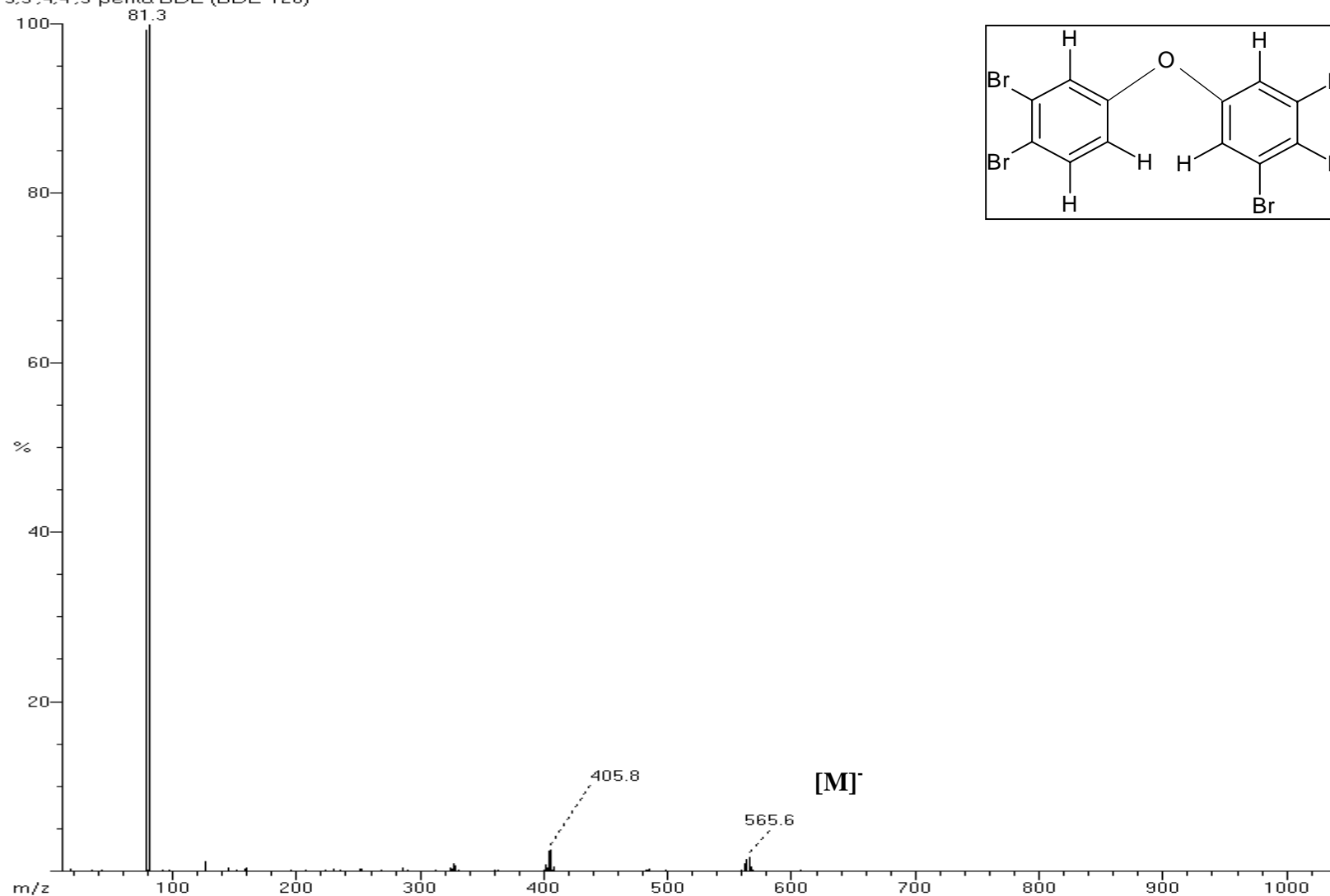
bde120 | bde120 450pg  
Scan: 426(406-420) TIC=2330130 Base=68.6%FS #ions=521 RT=13.67  
2,3',4,5,5'-penta BDE (BDE-120)



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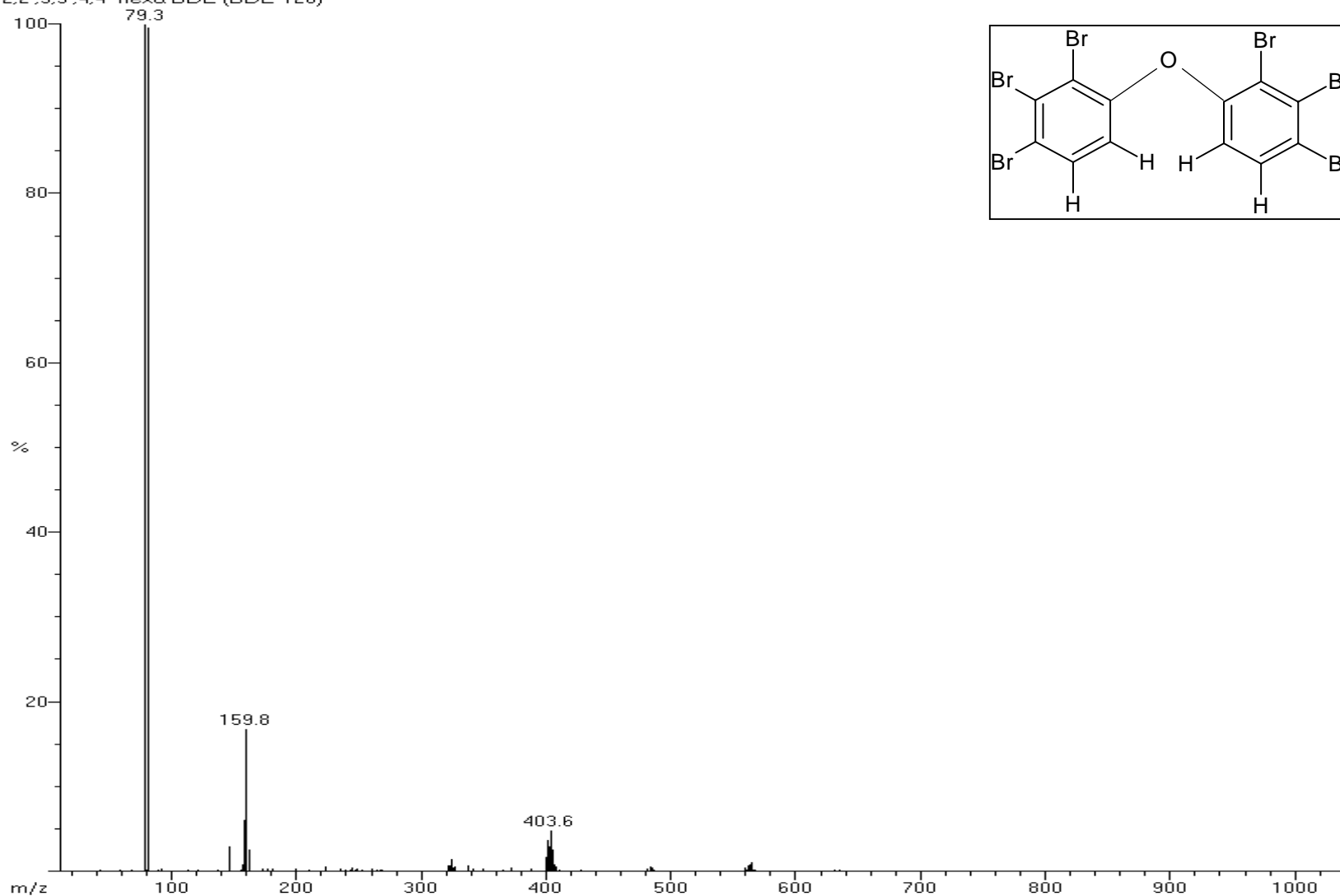
bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 480(450-464) TIC=1442400 Base=56.7%FS #ions=499 RT=14.77

3,3',4,4',5-penta BDE (BDE-126)



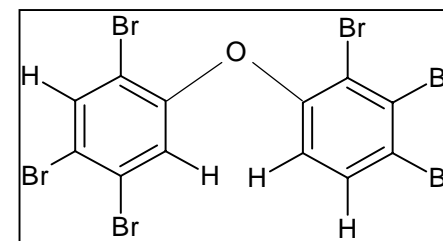
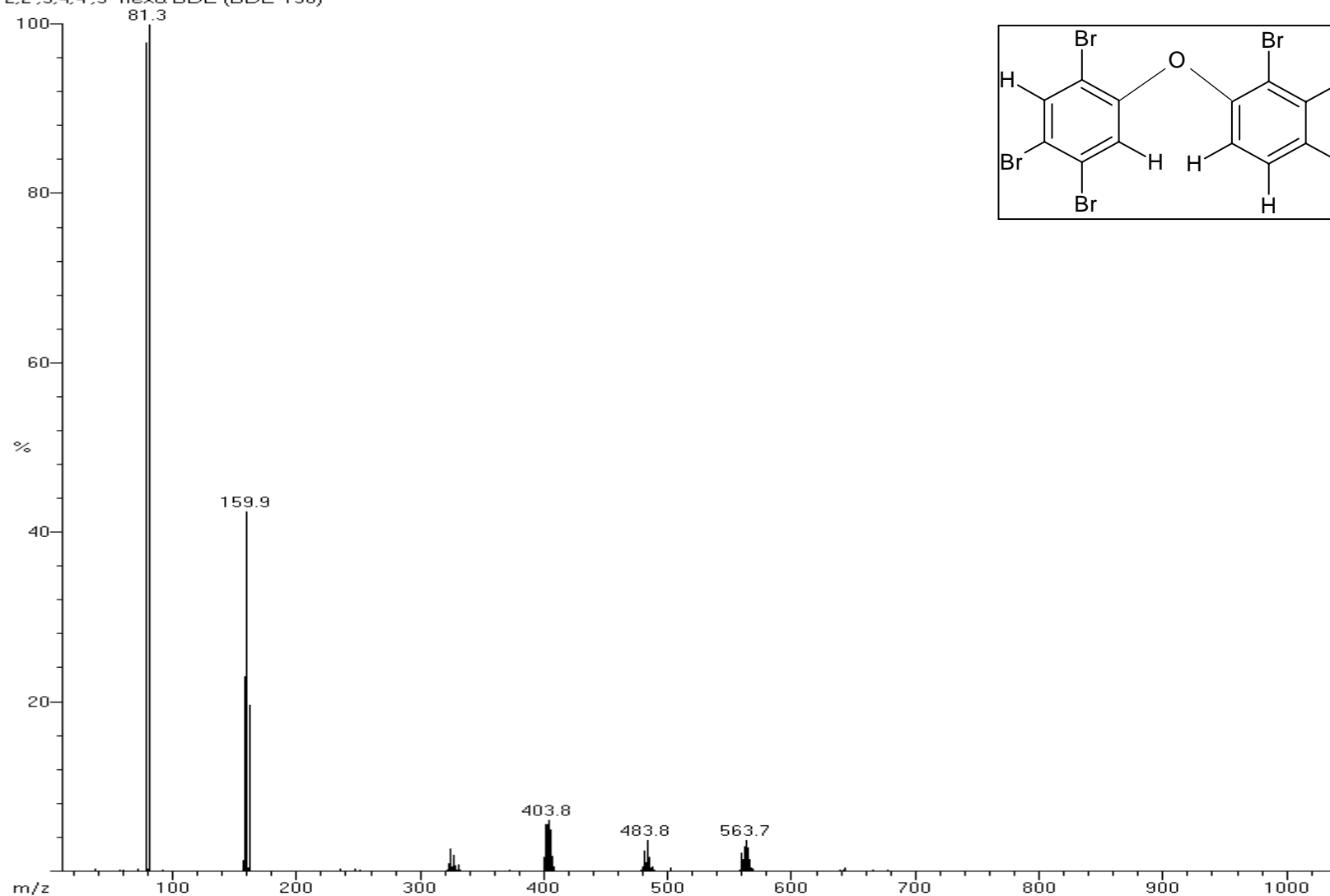
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bde128 | bde128 450pg  
Scan: 593(637-651) TIC=1226938 Base=39.9%FS #ions=412 RT=17.08  
2,2',3,3',4,4'-hexa BDE (BDE-128)



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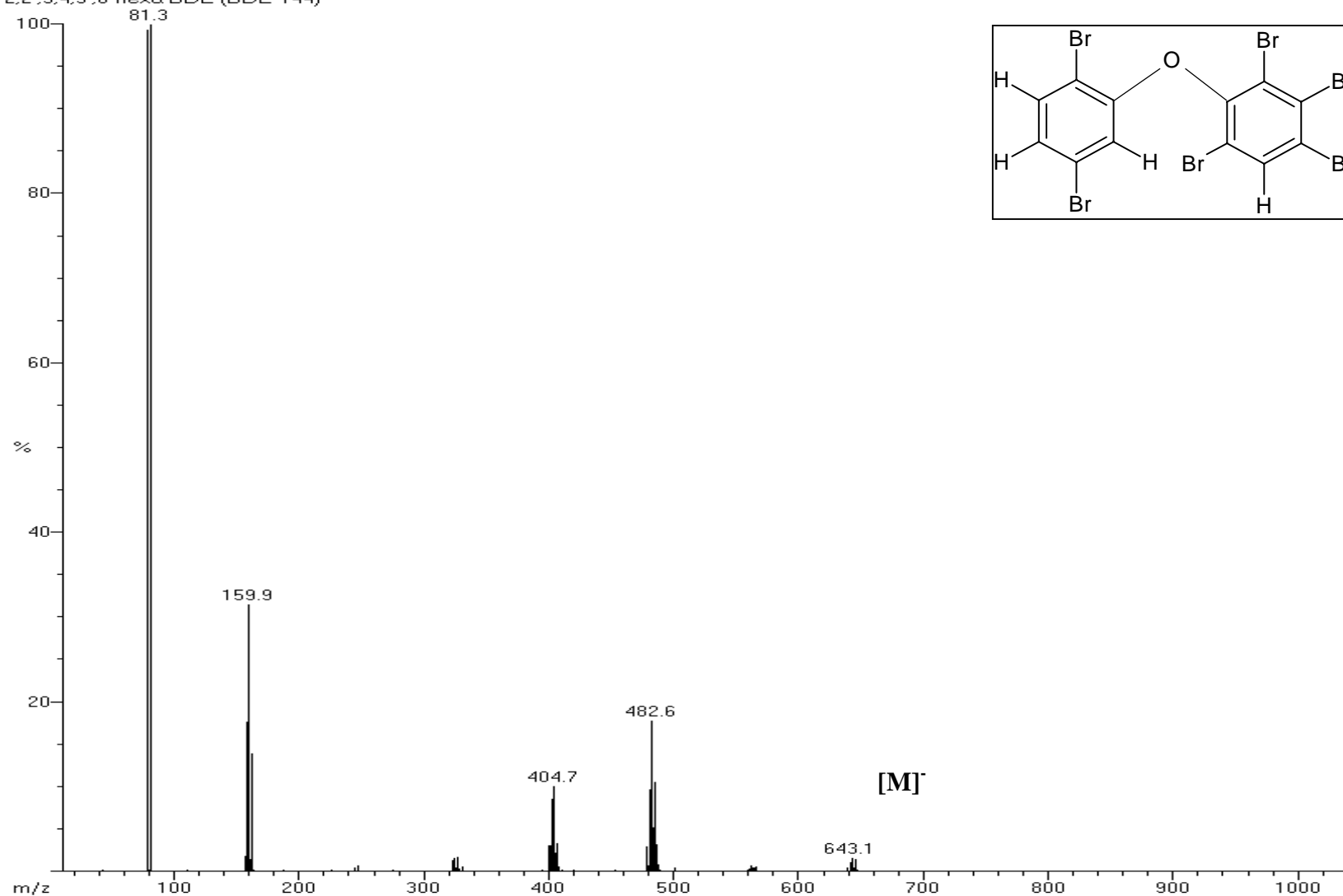
bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 555(503-517) TIC=2754172 Base=68.5%FS #ions=658 RT=16.3  
2,2',3,4,4',5'-hexa BDE (BDE-138)



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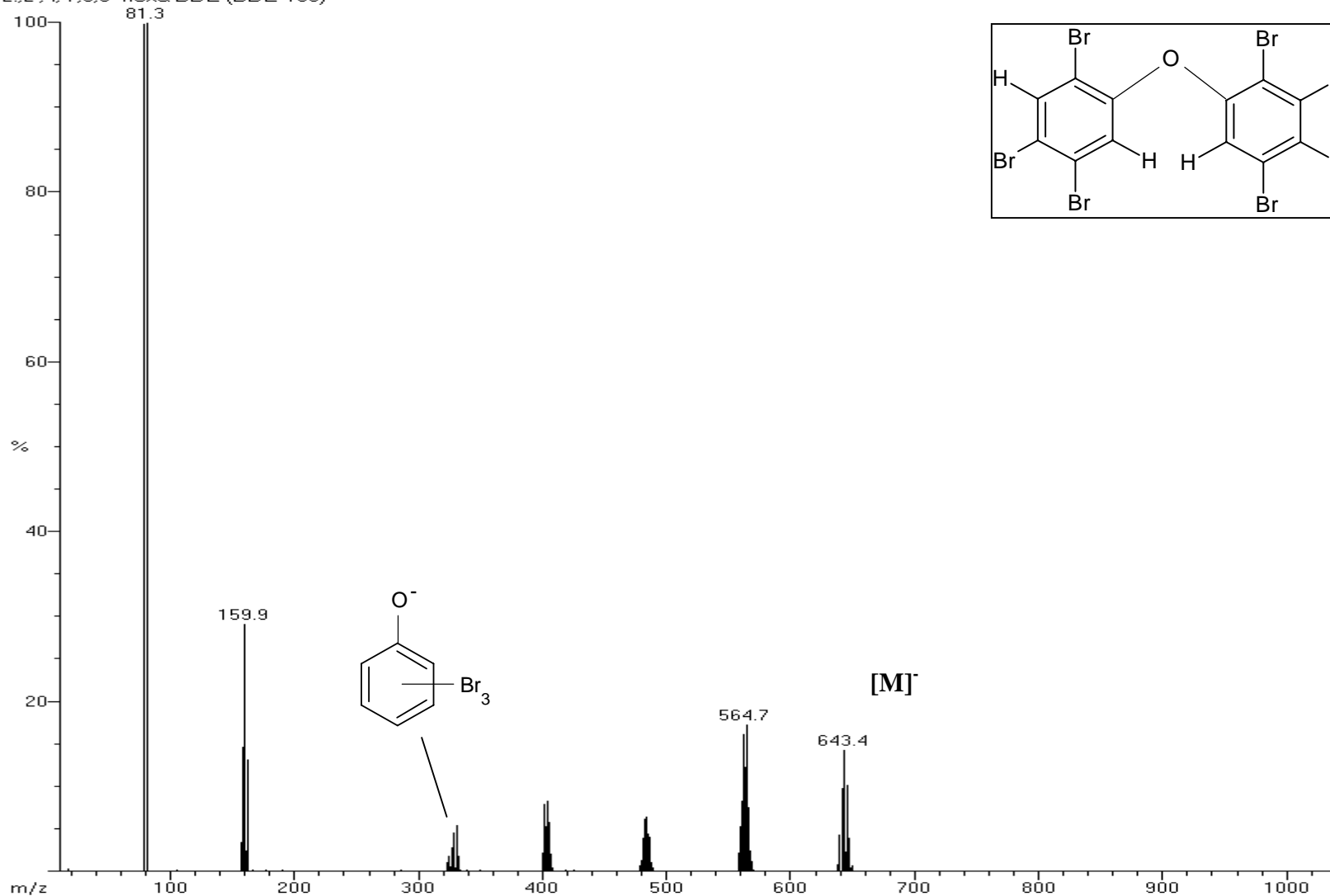
bde144 | bde144 450pg  
Scan: 502(446-460) TIC=2885828 Base=69.1%FS #ions=583 RT=15.22  
2,2',3,4,5',6-hexa BDE (BDE-144)



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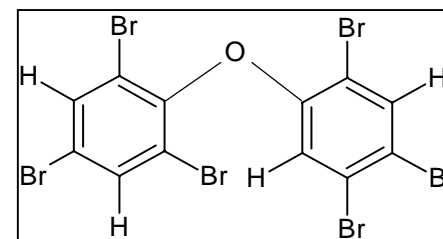
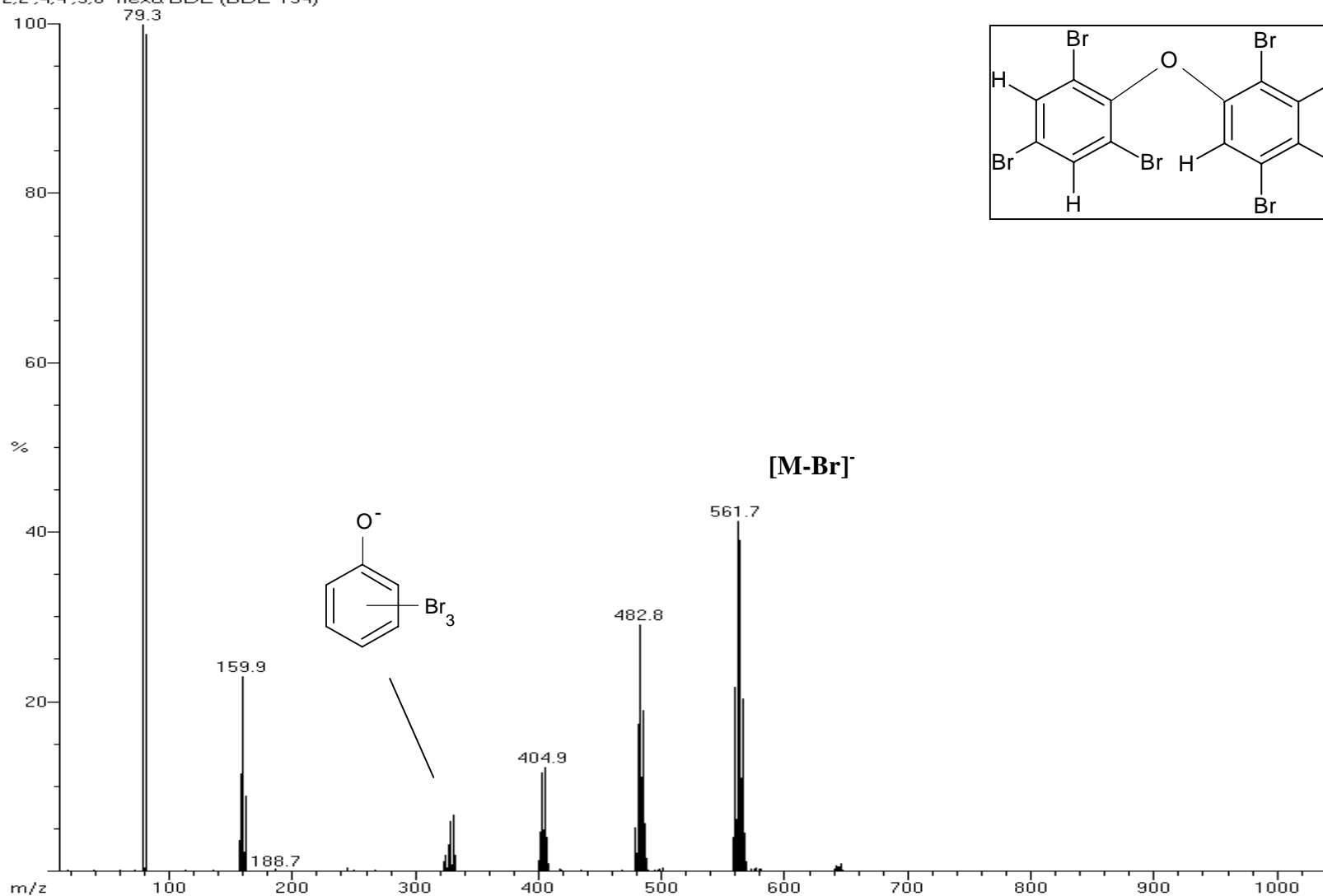
bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 520(450-464) TIC=4220576 Base=76.5%FS #ions=856 RT=15.58

2,2',4,4',5,5'-hexa BDE (BDE-153)



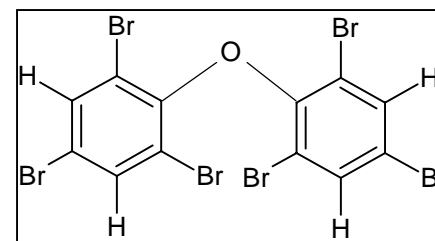
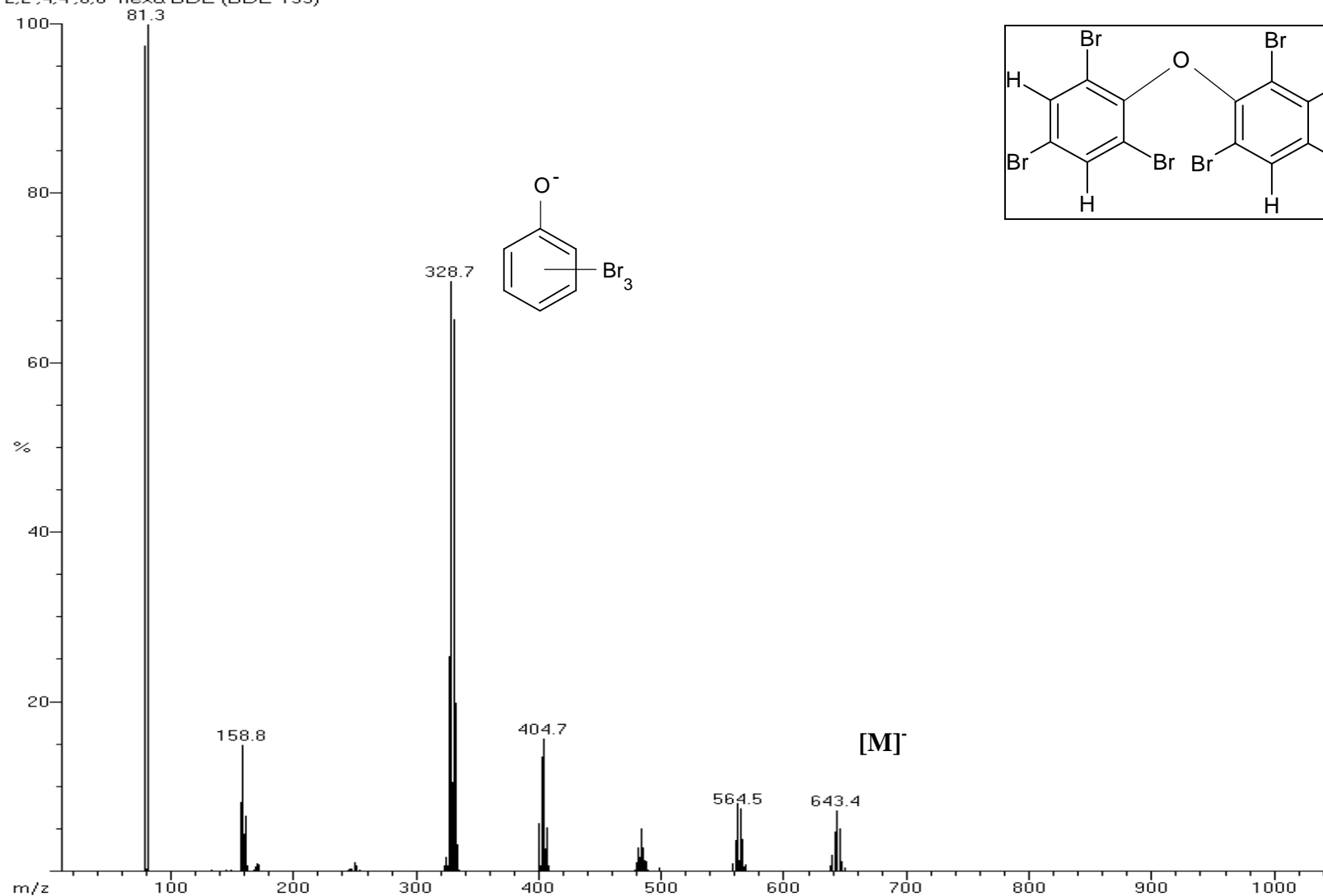
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bdemxe500\_1000\_2500ng.c | bdemxe 500/1000/2500ng @200C  
Scan: 493(450-464) TIC=4971639 Base=74.8%FS #ions=1143 RT=15.03  
2,2',4,4',5,6'-hexa BDE (BDE-154)

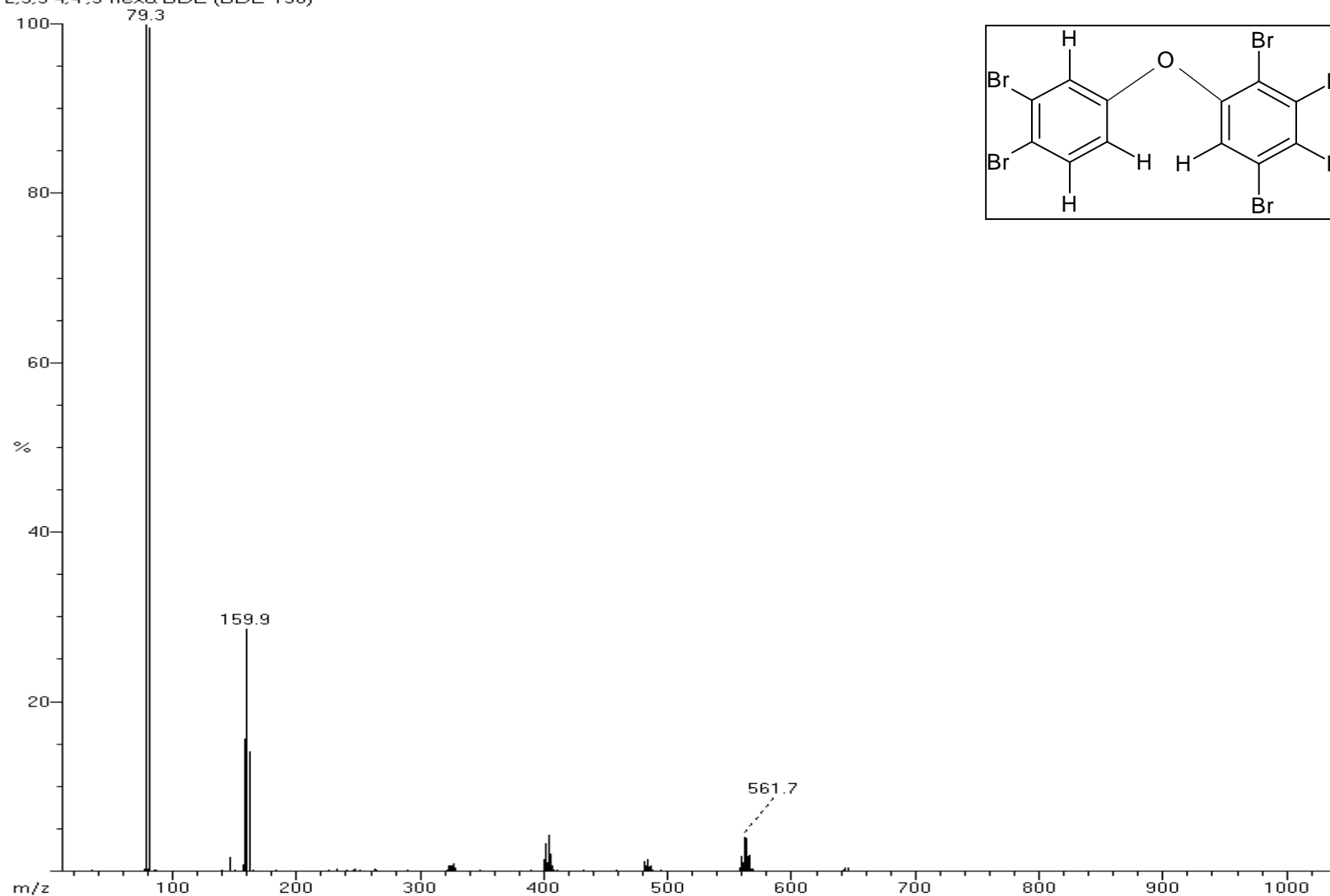


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bde155 | bde155 450pg  
Scan: 478(451-465) TIC=4345652 Base=70.3%FS #ions=704 RT=14.73  
2,2',4,4',6,6'-hexa BDE (BDE-155)

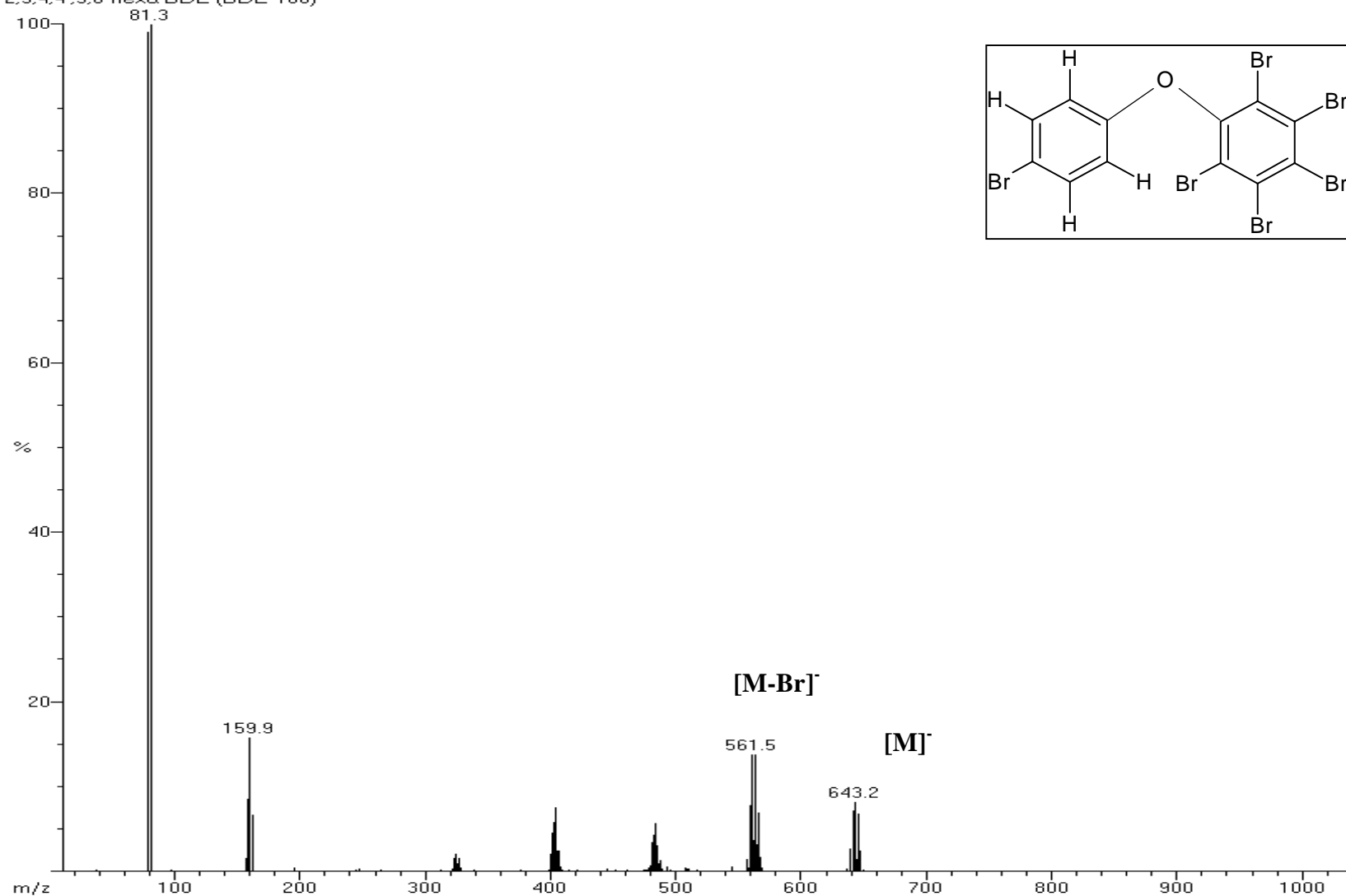


bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 569(503-517) TIC=2227484 Base=64.1%FS #ions=576 RT=16.58  
2,3,3',4,4',5-hexa BDE (BDE-156)



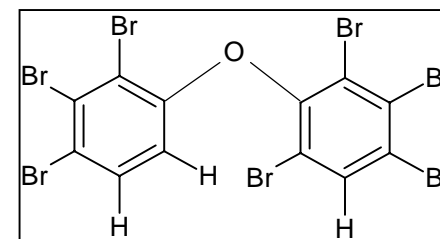
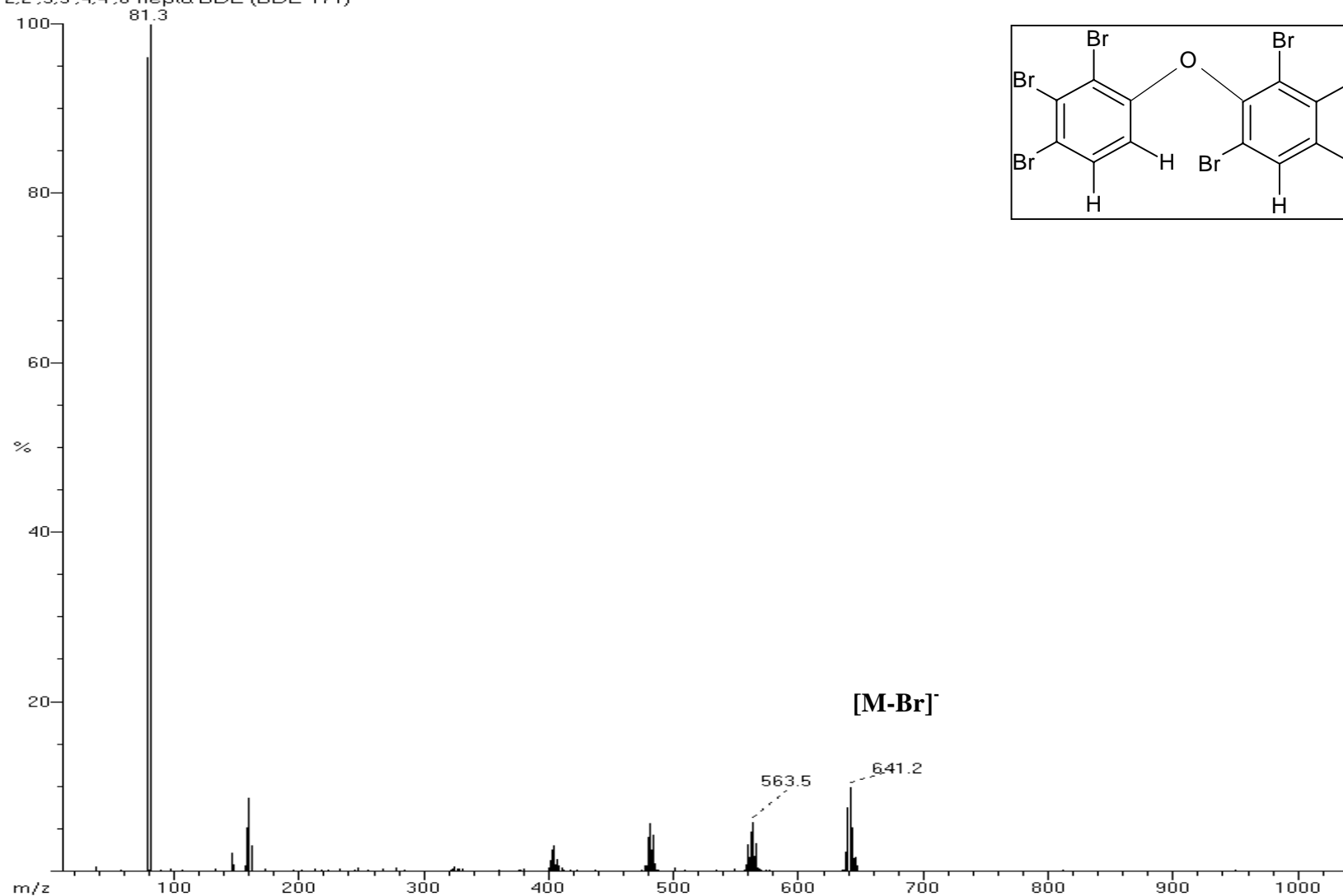
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bde166 | bde166 450pg  
Scan: 559(488-502) TIC=3337428 Base=77.1%FS #ions=973 RT=16.38  
2,3,4,4',5,6-hexa BDE (BDE-166)



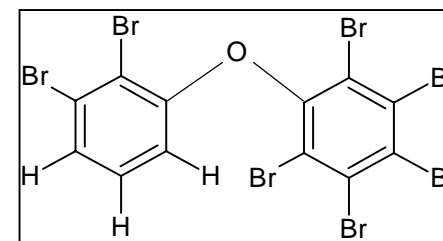
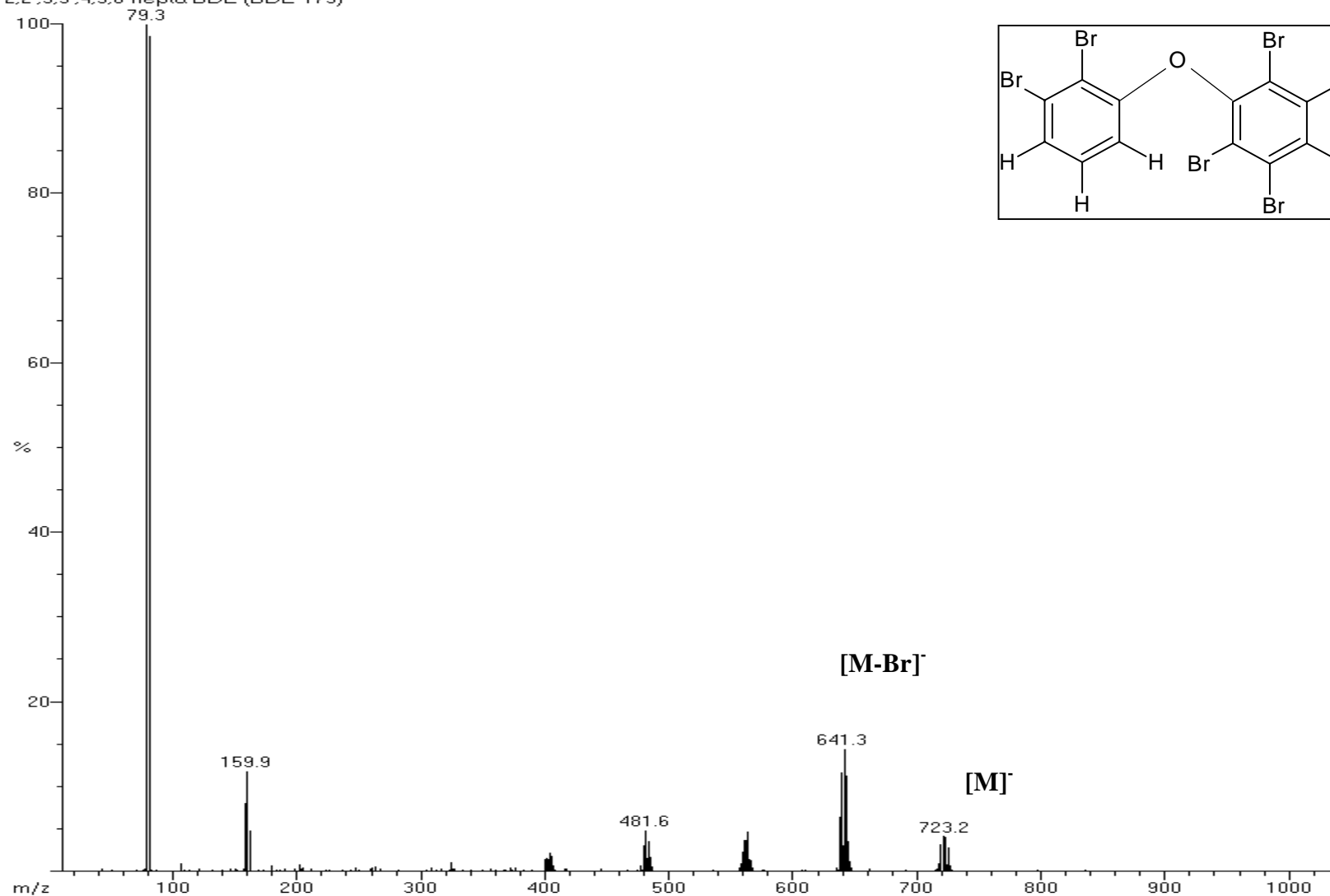
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bde171 | bde171 1000pg  
Scan: 645(705-719) TIC=1514773 Base=42%FS #ions=411 RT=18.13  
2,2',3,3',4,4',6-hepta BDE (BDE-171)



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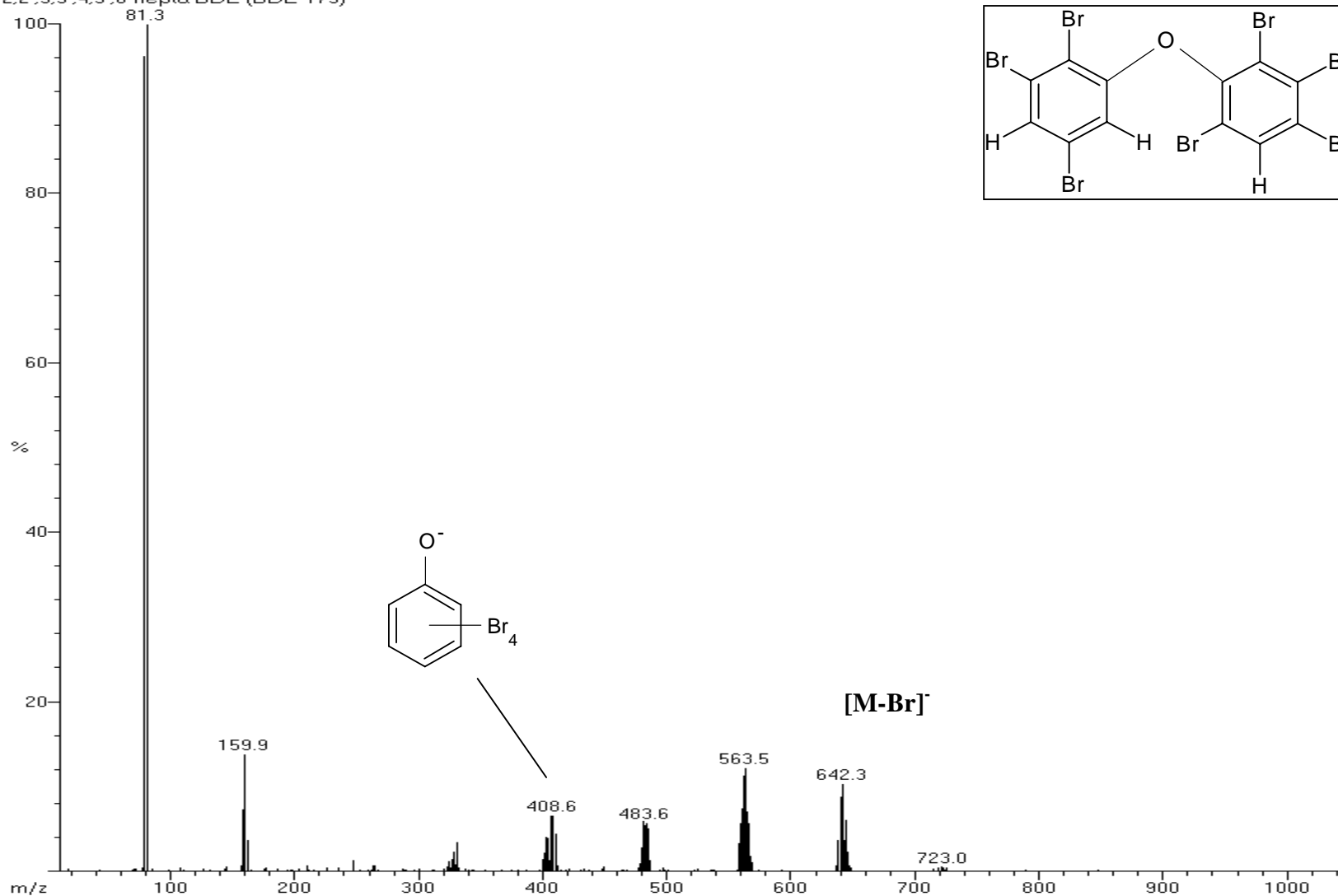
bde173 | bde173 450pg  
Scan: 645(705-719) TIC=1261267 Base=30.8%FS #ions=424 RT=18.13  
2,2',3,3',4,5,6-hepta BDE (BDE-173)



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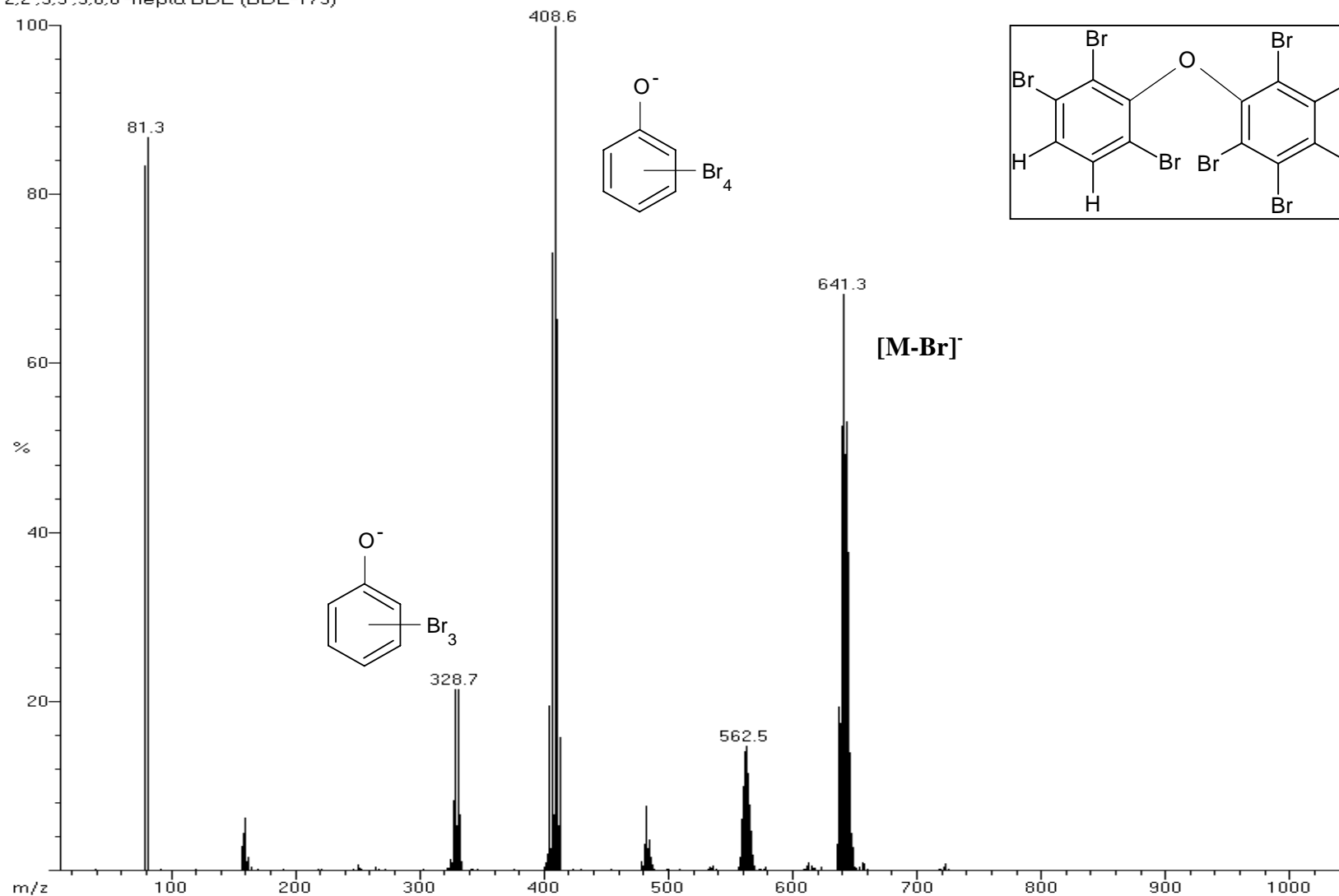


bde175 | bde175 450pg  
Scan: 598(656-670) TIC=1514498 Base=31.2%FS #ions=485 RT=17.18  
2,2',3,3',4,5',6-hepta BDE (BDE-175)



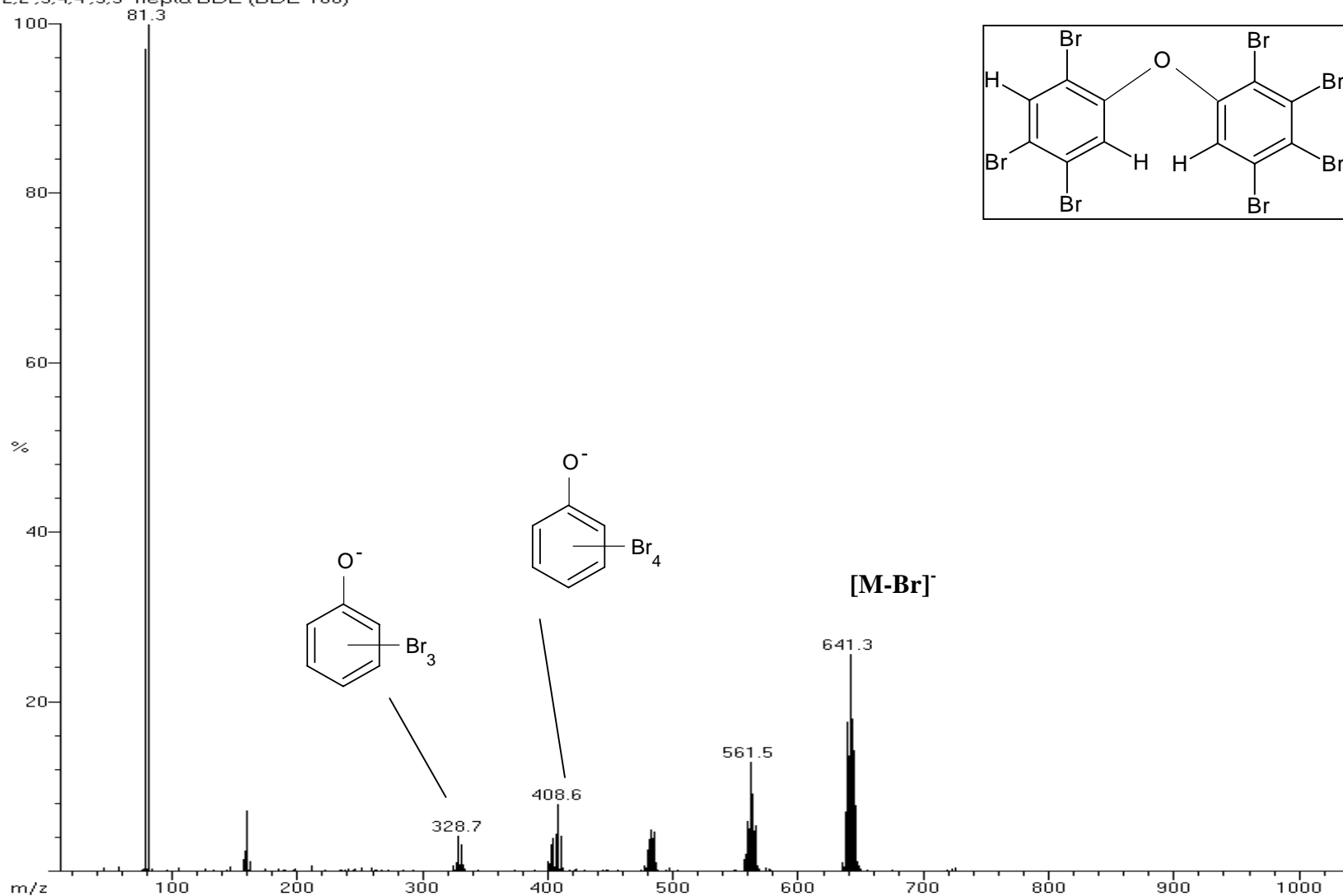
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bde179 | bde179 500pg  
Scan: 589(515-529) TIC=7678895 Base=65.1%FS #ions=1345 RT=17  
2,2',3,3',5,6,6'-hepta BDE (BDE-179)



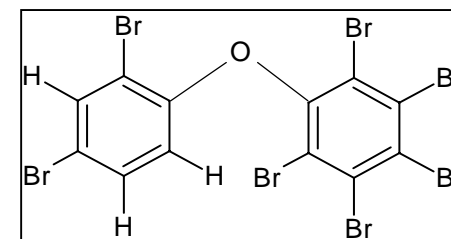
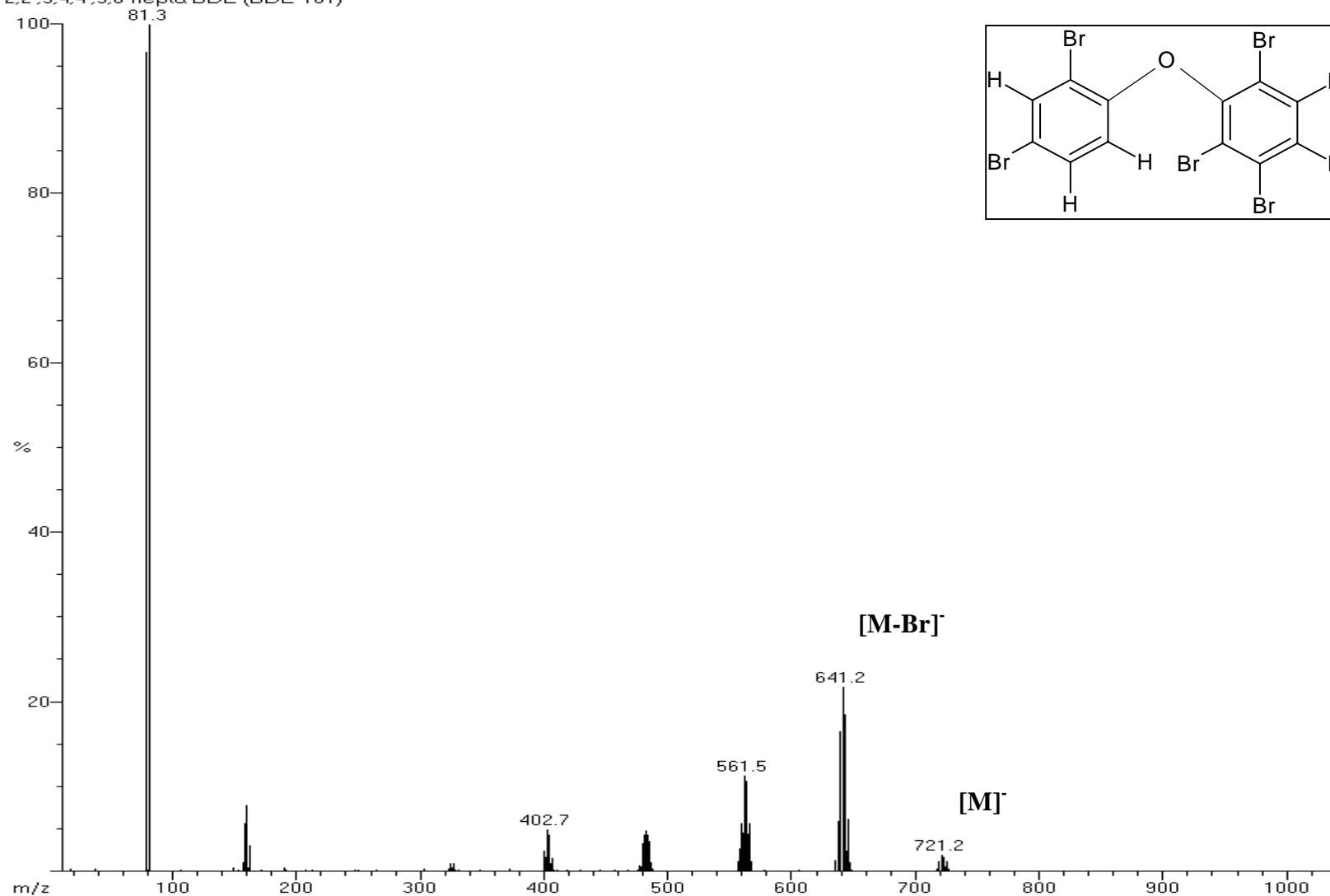
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bde180 | bde180 450pg  
Scan: 604(661-675) TIC=1908958 Base=35.5%FS #ions=437 RT=17.3  
2,2',3,4,4',5,5'-hepta BDE (BDE-180)

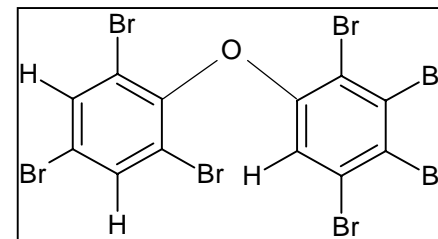
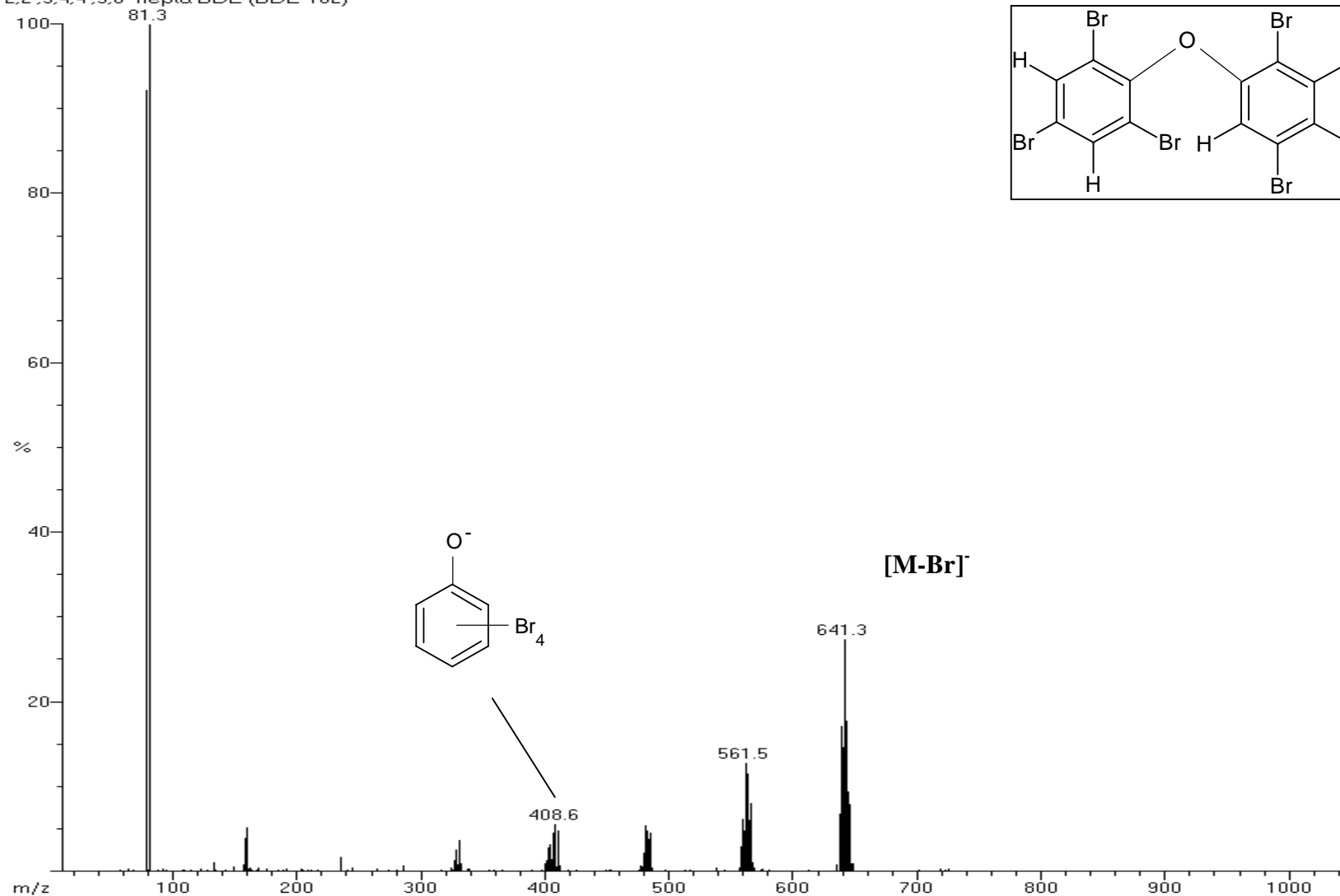


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bde181 | bde181 450pg  
Scan: 638(695-709) TIC=2297974 Base=50.7%FS #ions=466 RT=18  
2,2',3,4,4',5,6-hepta BDE (BDE-181)

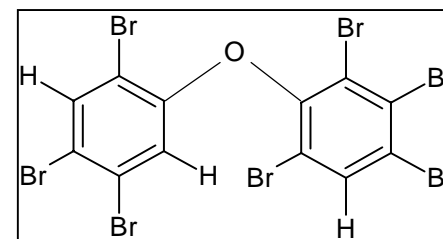
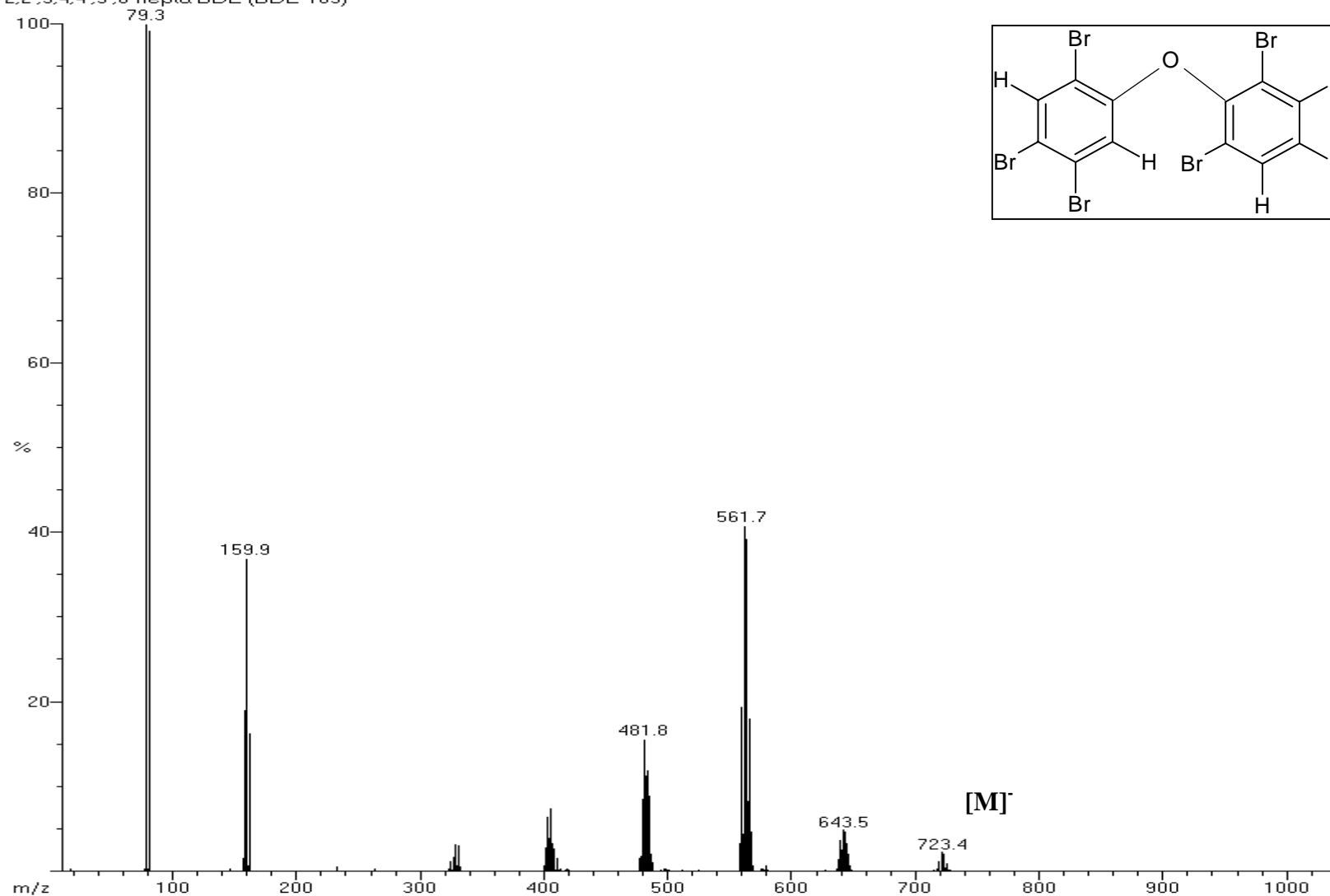


bde182 | bde182 450pg  
Scan: 605(660-674) TIC=1935806 Base=36.3%FS #ions=513 RT=17.32  
2,2',3,4,4',5,6'-hepta BDE (BDE-182)



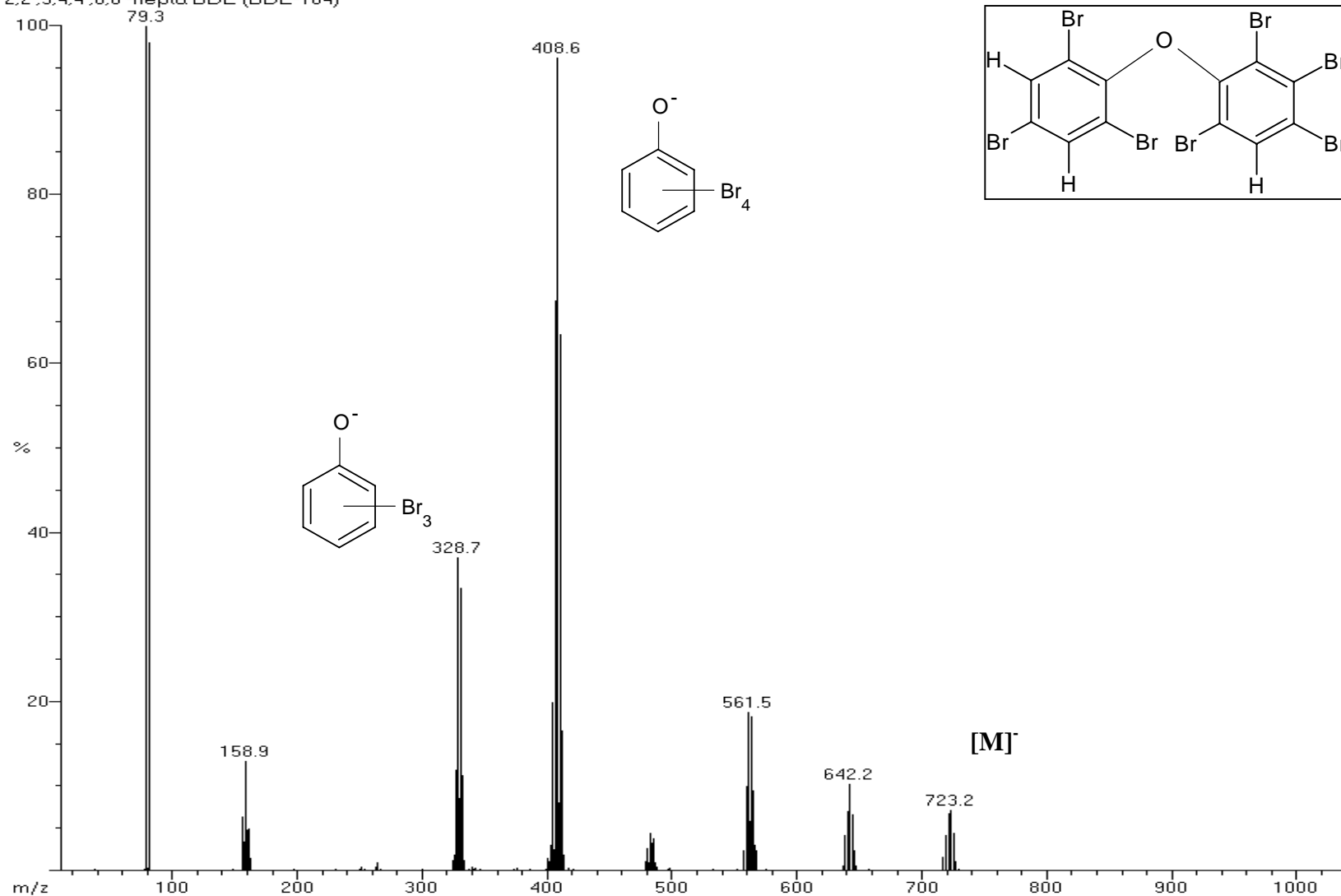
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bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 597(651-665) TIC=4606653 Base=71%FS #ions=1023 RT=17.15  
2,2',3,4,4',5',6'-hepta BDE (BDE-183)



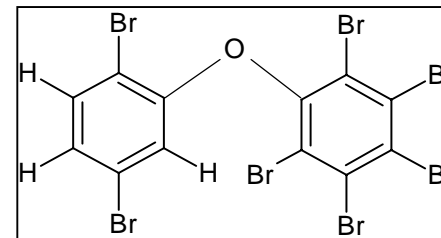
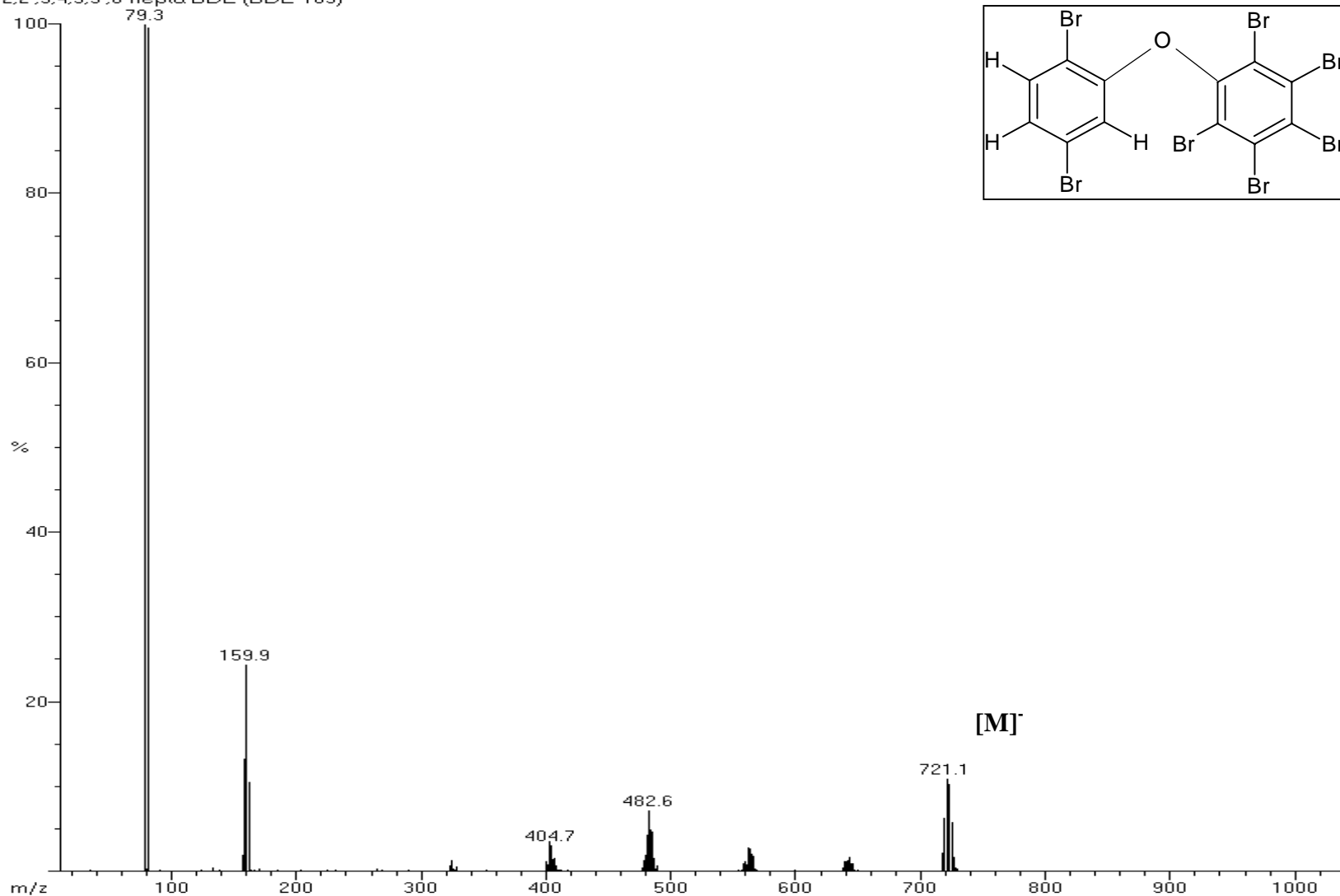
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bde184 | bde184 500pg  
Scan: 584(514-528) TIC=5545092 Base=63%FS #ions=757 RT=16.9  
2,2',3,4,4',6,6'-hepta BDE (BDE-184)



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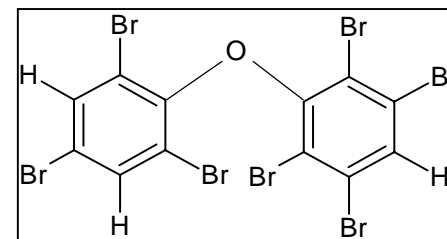
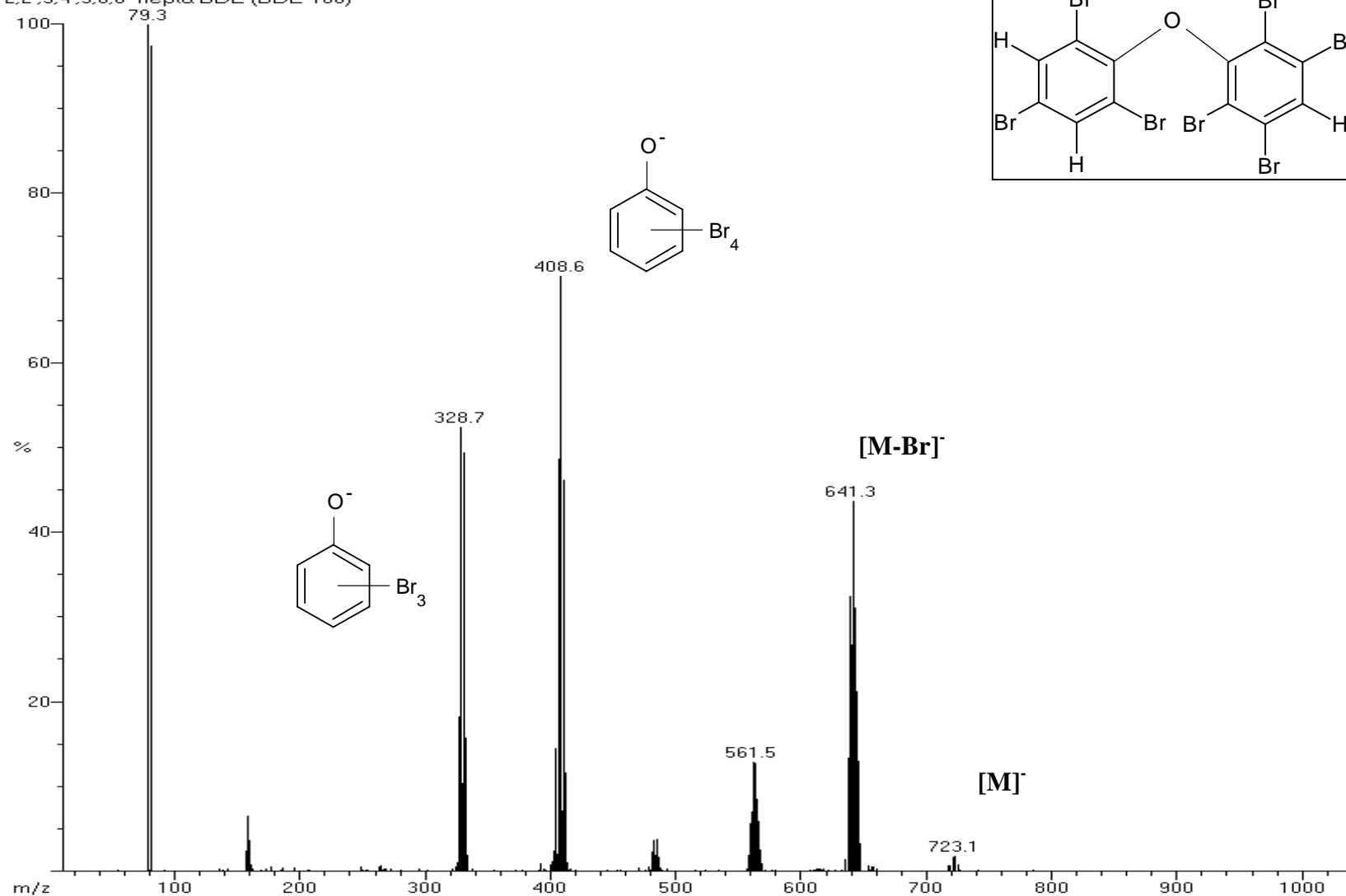
bde185 | bde185 500pg  
Scan: 607(667-681) TIC=2397187 Base=58.6%FS #ions=555 RT=17.37  
2,2',3,4,5,5',6-hepta BDE (BDE-185)



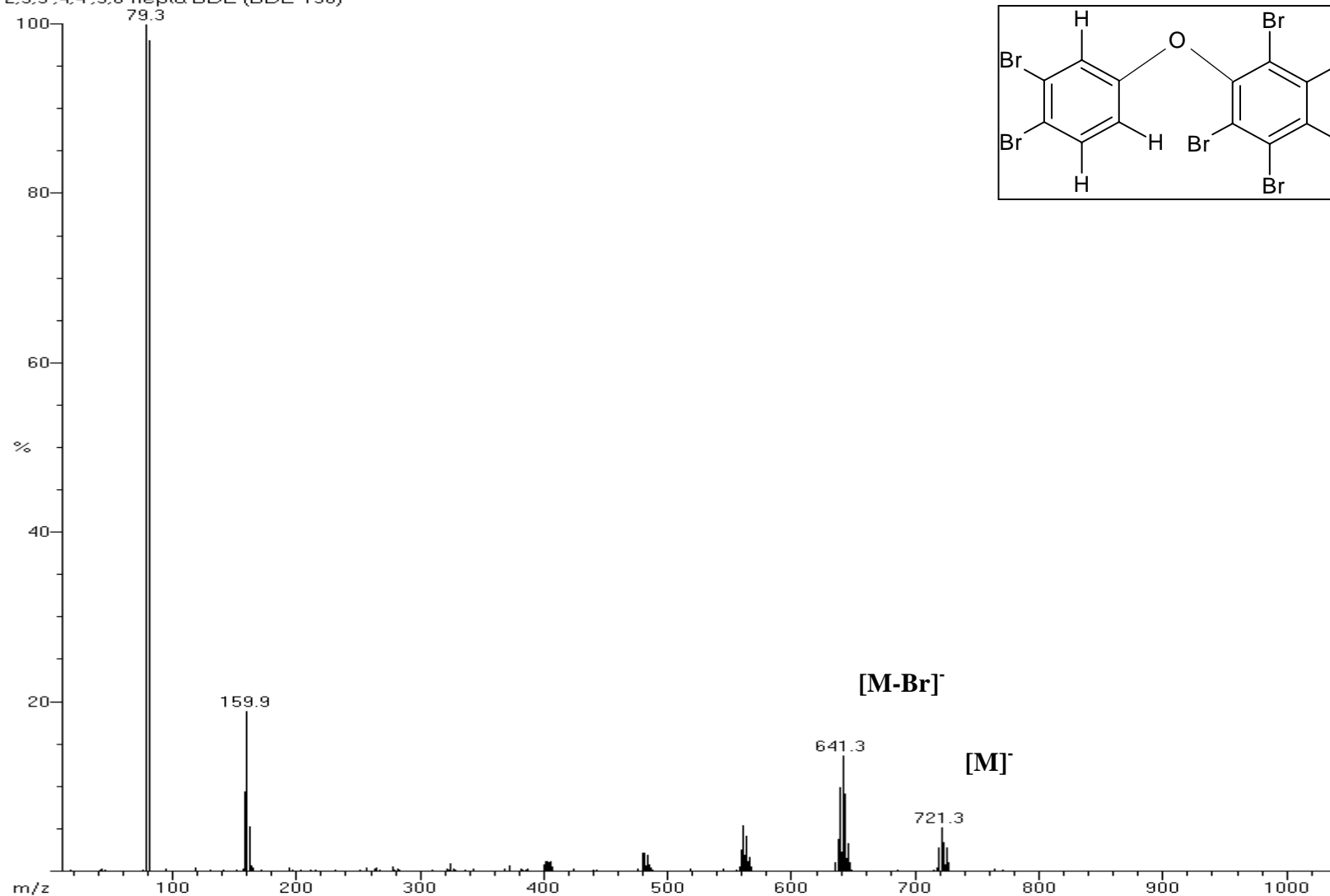
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bde188 | bde188 500pg  
Scan: 577(506-520) TIC=4515619 Base=44%FS #ions=745 RT=16.75  
2,2',3,4',5,6,6'-hepta BDE (BDE-188)

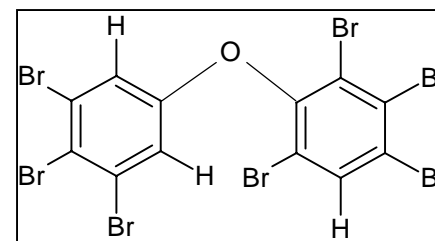
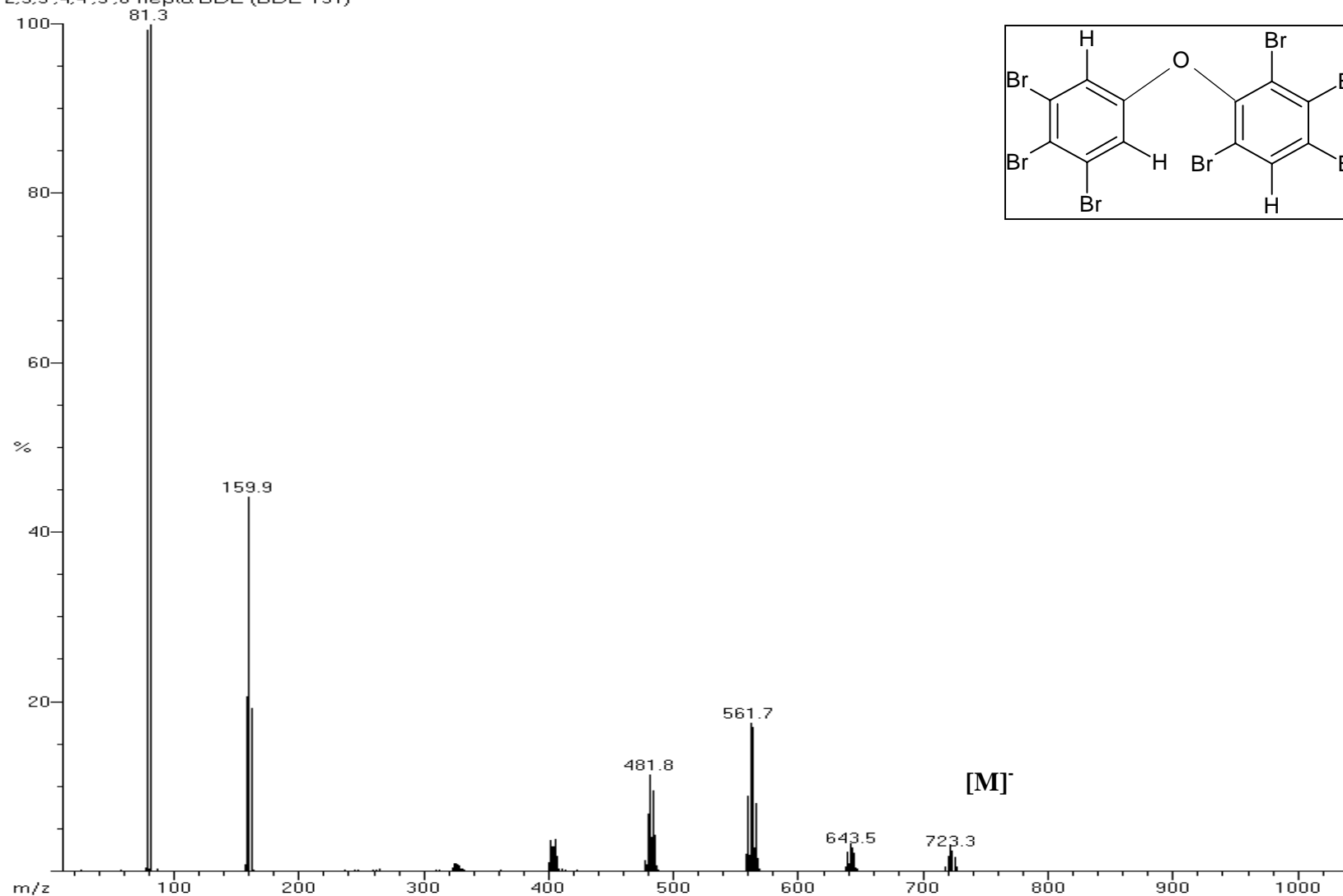


bde190 | bde190 500pg  
Scan: 645(703-717) TIC=1254157 Base=32.3%FS #ions=385 RT=18.13  
2,3,3',4,4',5,6-hepta BDE (BDE-190)



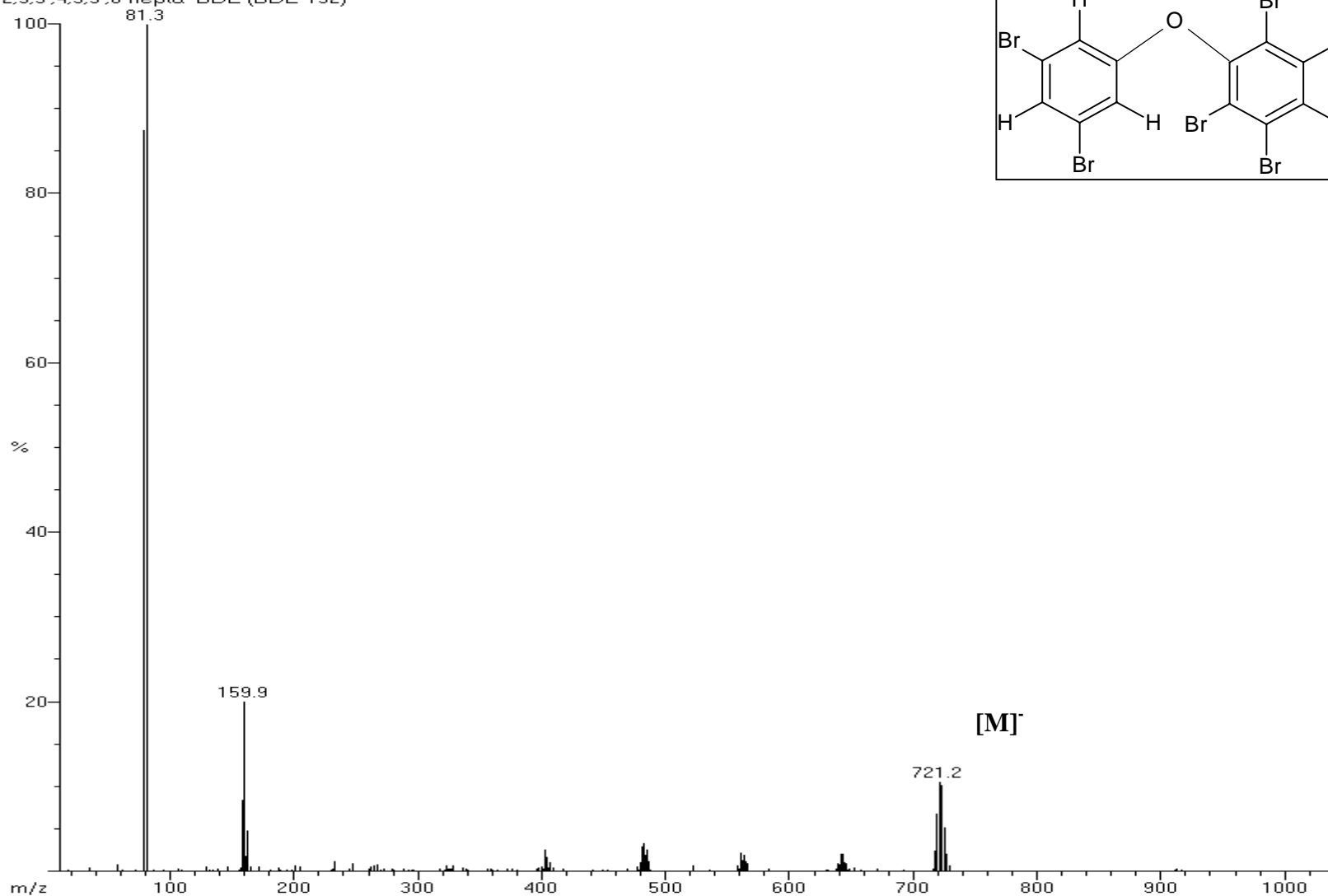
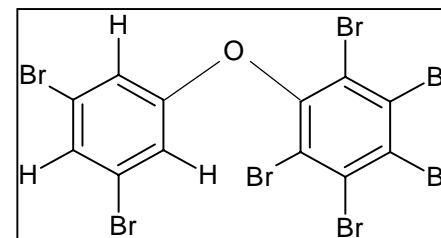
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bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 617(663-677) TIC=3522444 Base=71%FS #ions=704 RT=17.57  
2,3,3',4,4',5',6'-hepta BDE (BDE-191)



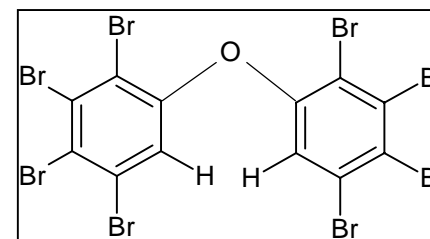
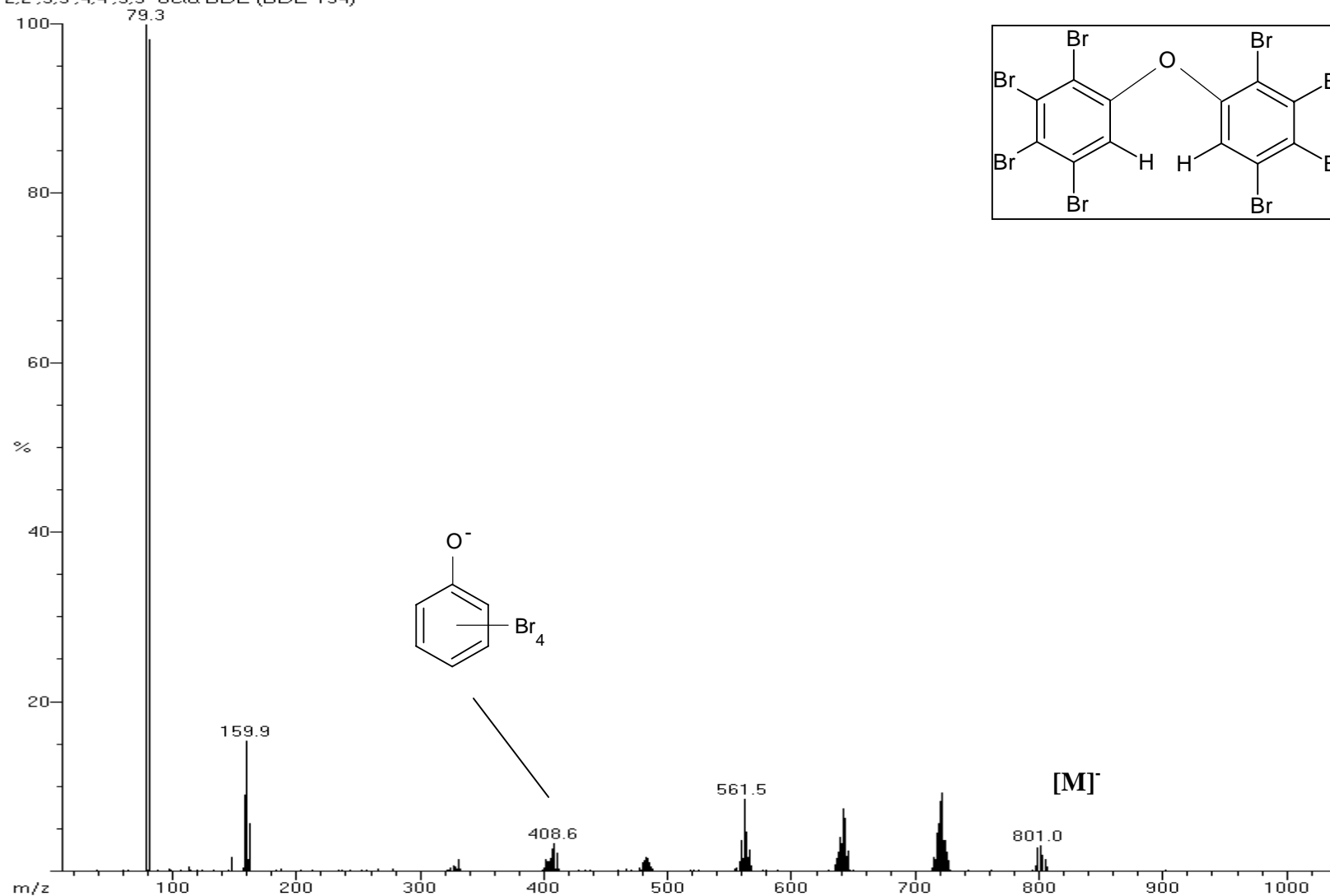
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bde192 | bde194 500pg  
Scan: 613(670-684) TIC=992705 Base=26.2%FS #ions=401 RT=17.48  
2,3,3',4,5,5',6-hepta-BDE (BDE-192)

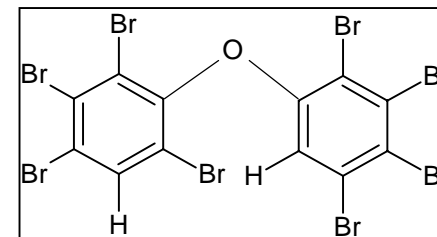
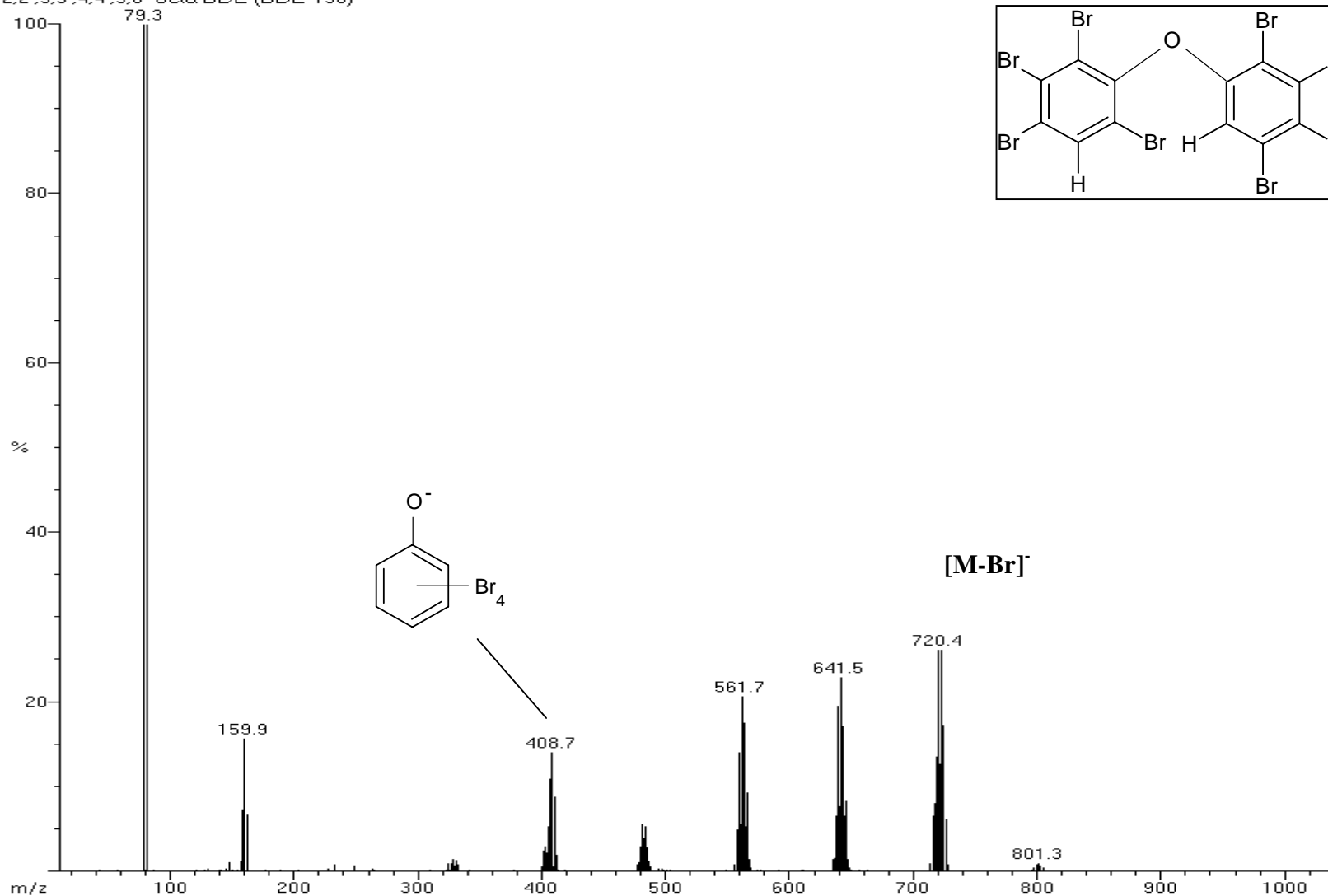


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bde194 | bde194 450pg  
Scan: 732(713-727) TIC=1598891 Base=35.9%FS #ions=449 RT=19.92  
2,2',3,3',4,4',5,5'-octa BDE (BDE-194)

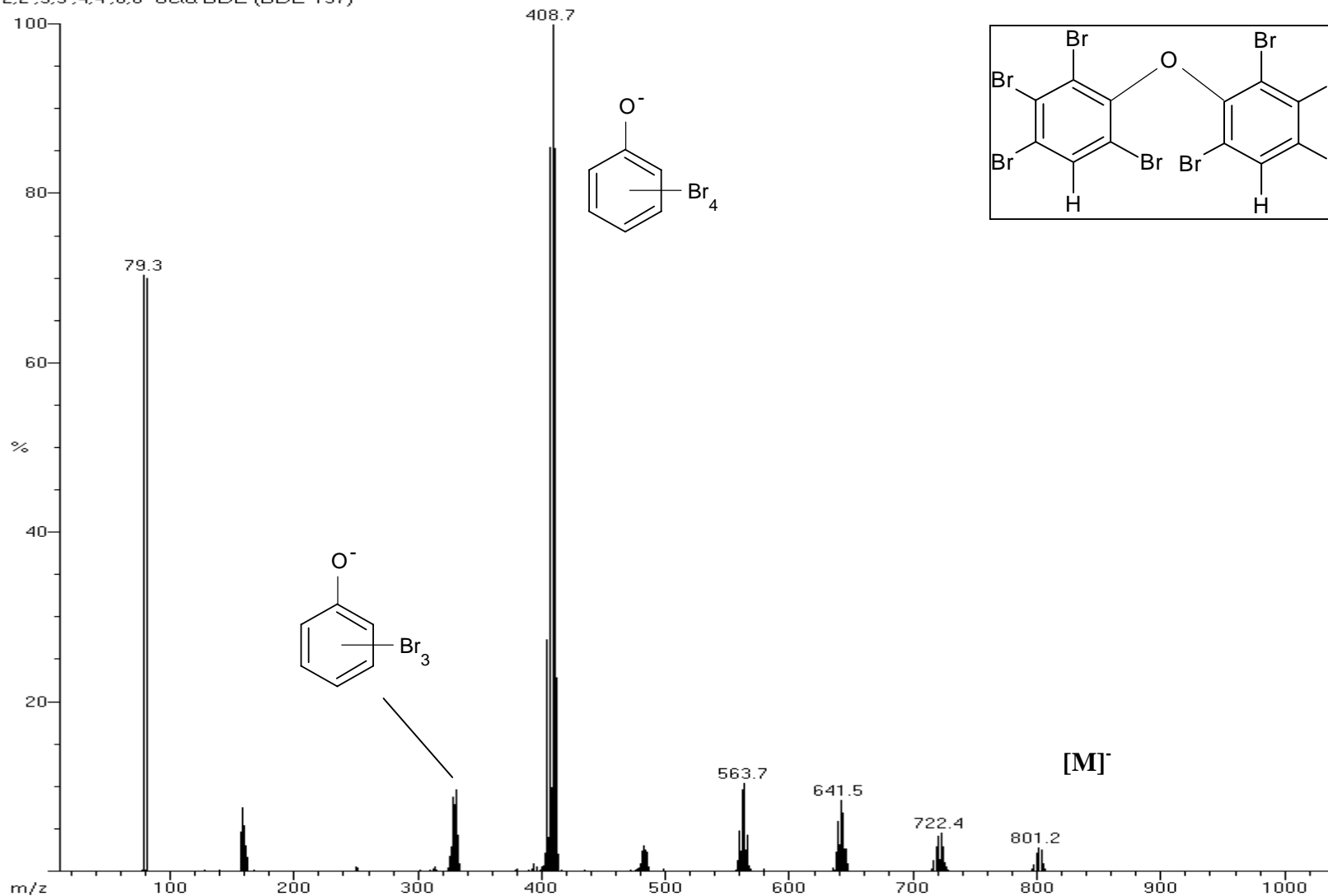


bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 702(753-767) TIC=4461488 Base=60.4%FS #ions=773 RT=19.3  
2,2',3,3',4,4',5,6'-octa BDE (BDE-196)



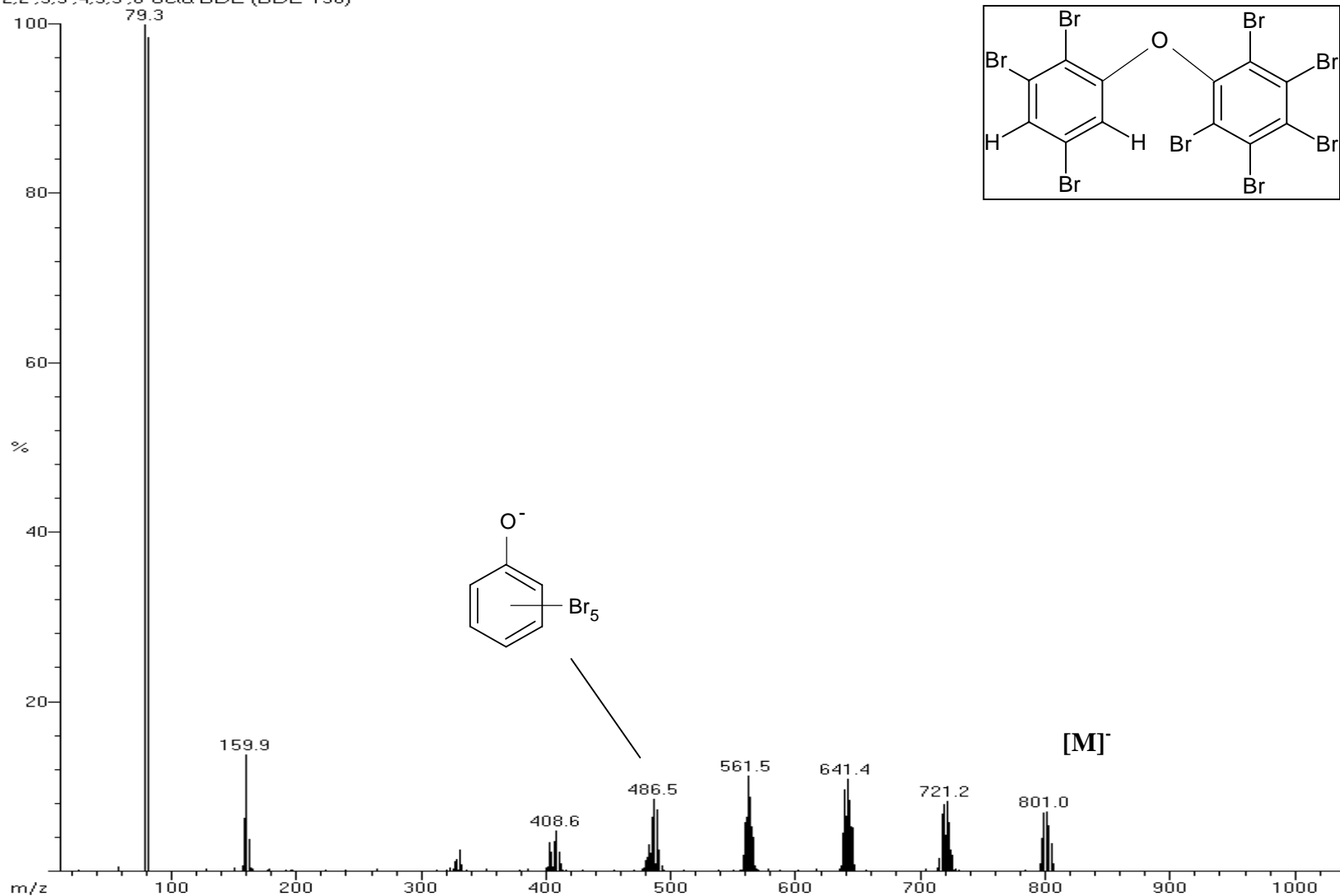
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bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 684(744-758) TIC=7252418 Base=94.2%FS #ions=1120 RT=18.93  
2,2',3,3',4,4',6,6'-octa BDE (BDE-197)



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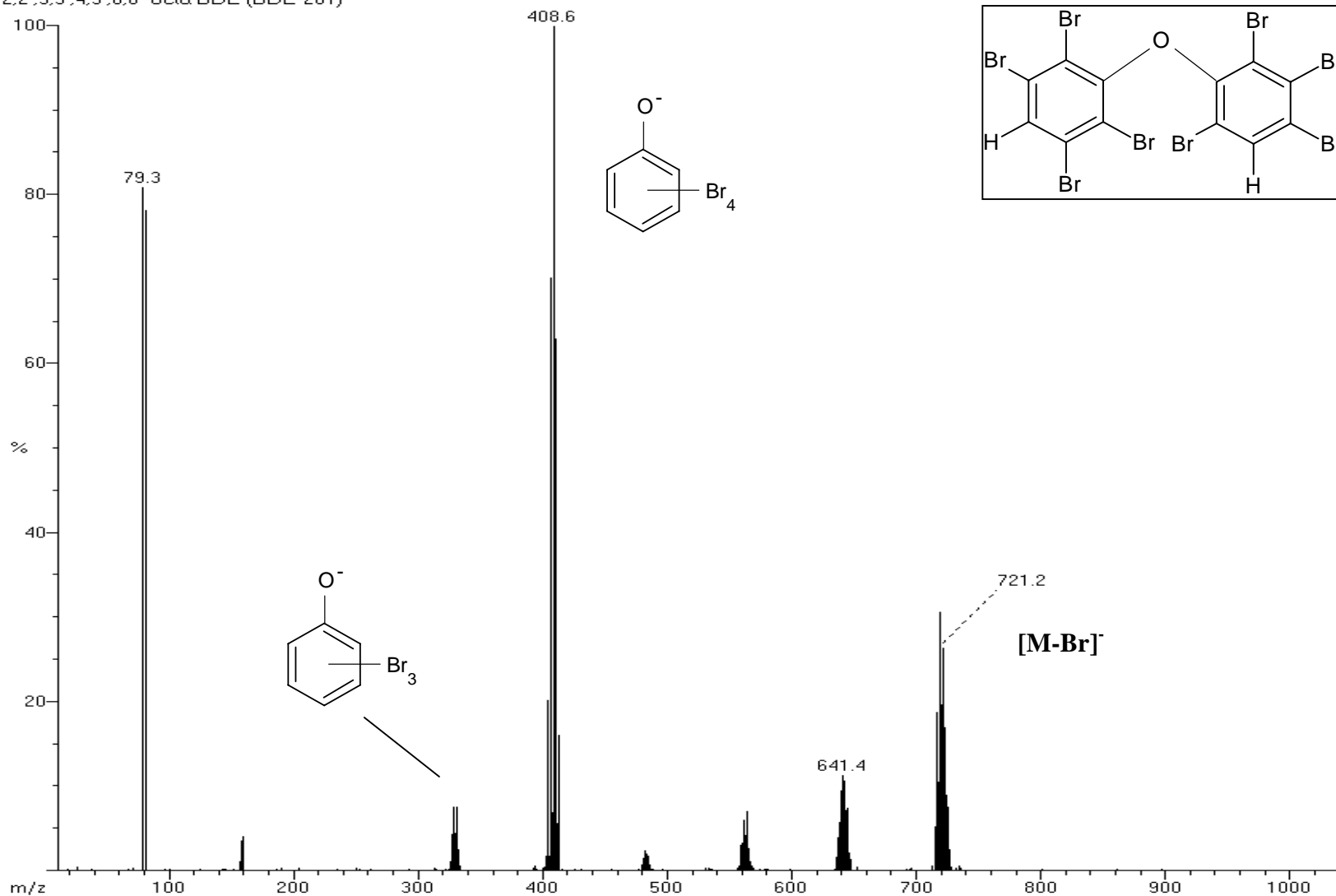
bde198 | bde198 450pg  
Scan: 696(740-754) TIC=2143306 Base=38.7%FS #ions=520 RT=19.18  
2,2',3,3',4,5,5',6-octa BDE (BDE-198)



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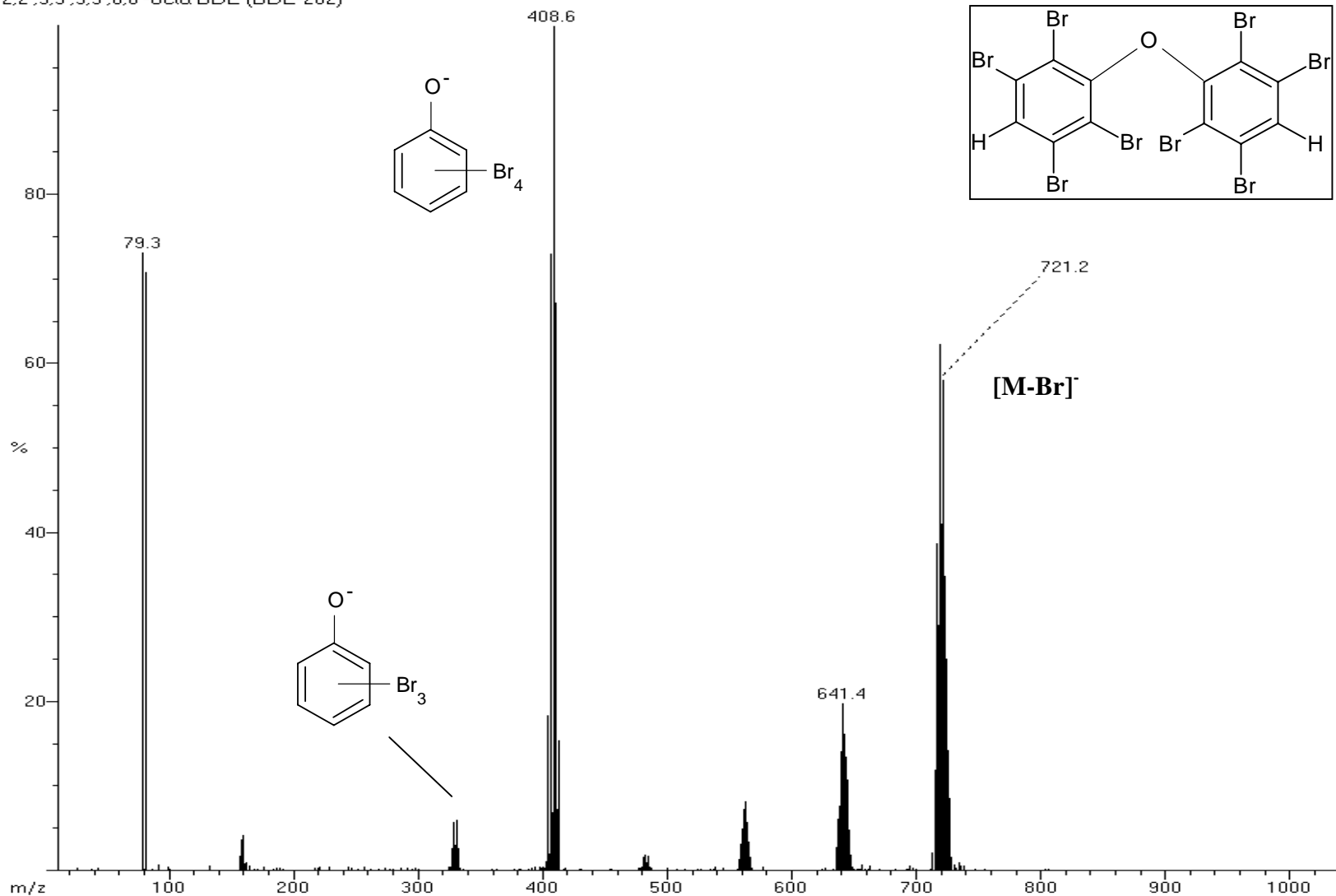


bde201 | bde202 450pg  
Scan: 678(625-639) TIC=3714497 Base=42.9%FS #ions=620 RT=18.8  
2,2',3,3',4,5',6,6'-octa BDE (BDE-201)



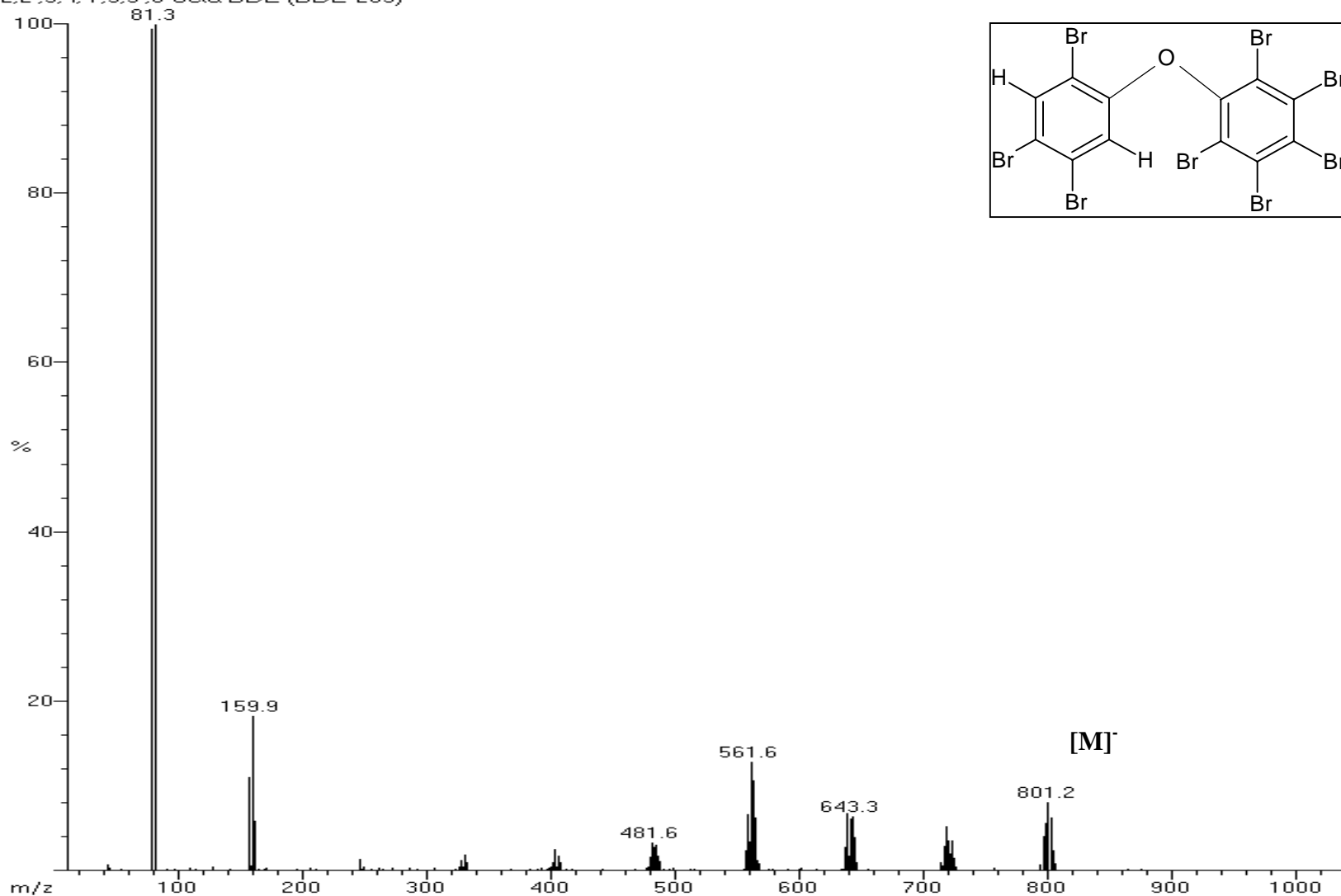
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bde202 | bde202 500pg  
Scan: 670(730-744) TIC=4003086 Base=35.2%FS #ions=606 RT=18.65  
2,2',3,3',5,5',6,6'-octa BDE (BDE-202)



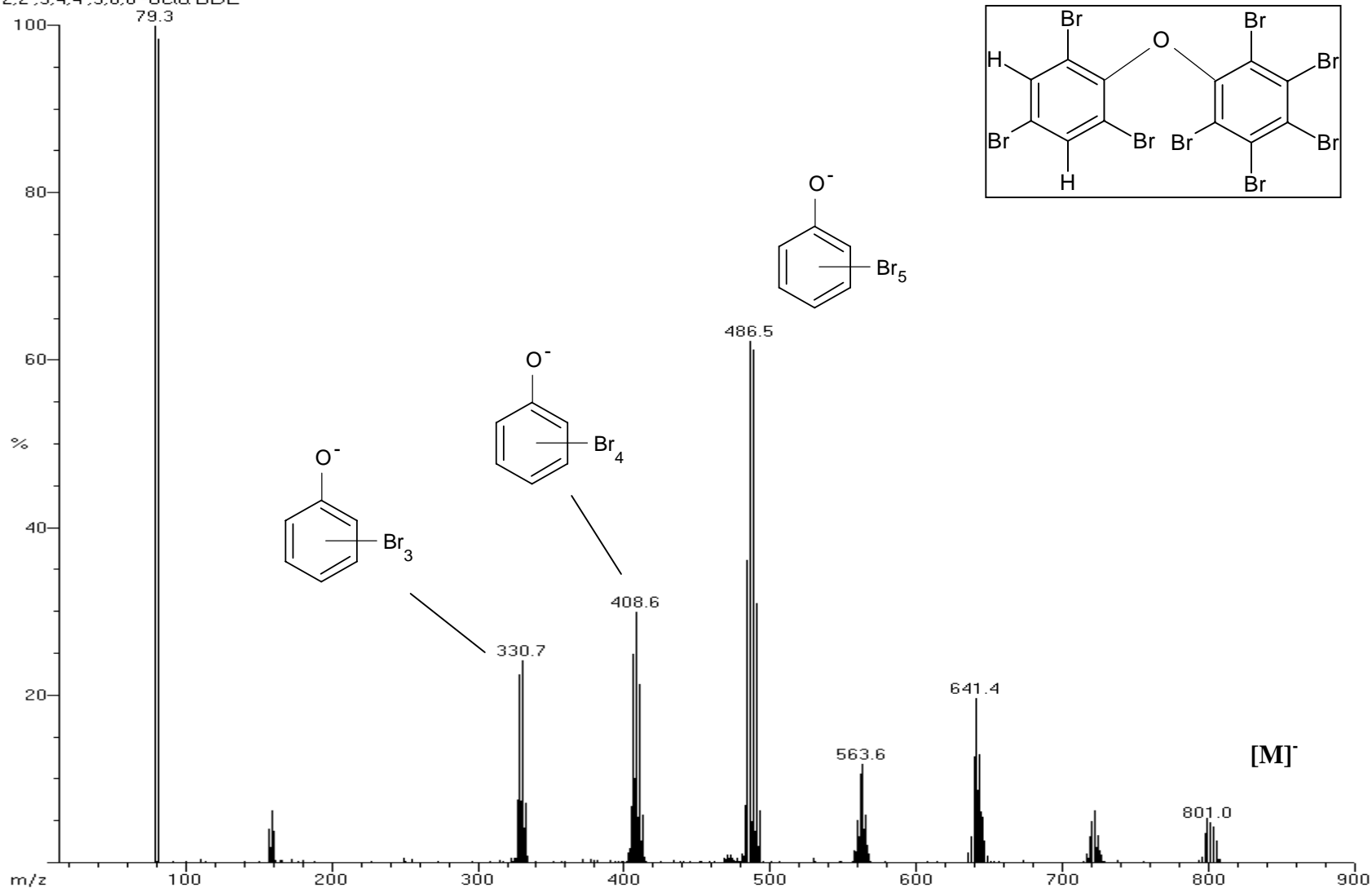
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bde203 | bde203 450pg  
Scan: 698(749-763) TIC=1474127 Base=31.4%FS #ions=463 RT=19.22  
2,2',3,4,4',5,5',6-octa BDE (BDE-203)



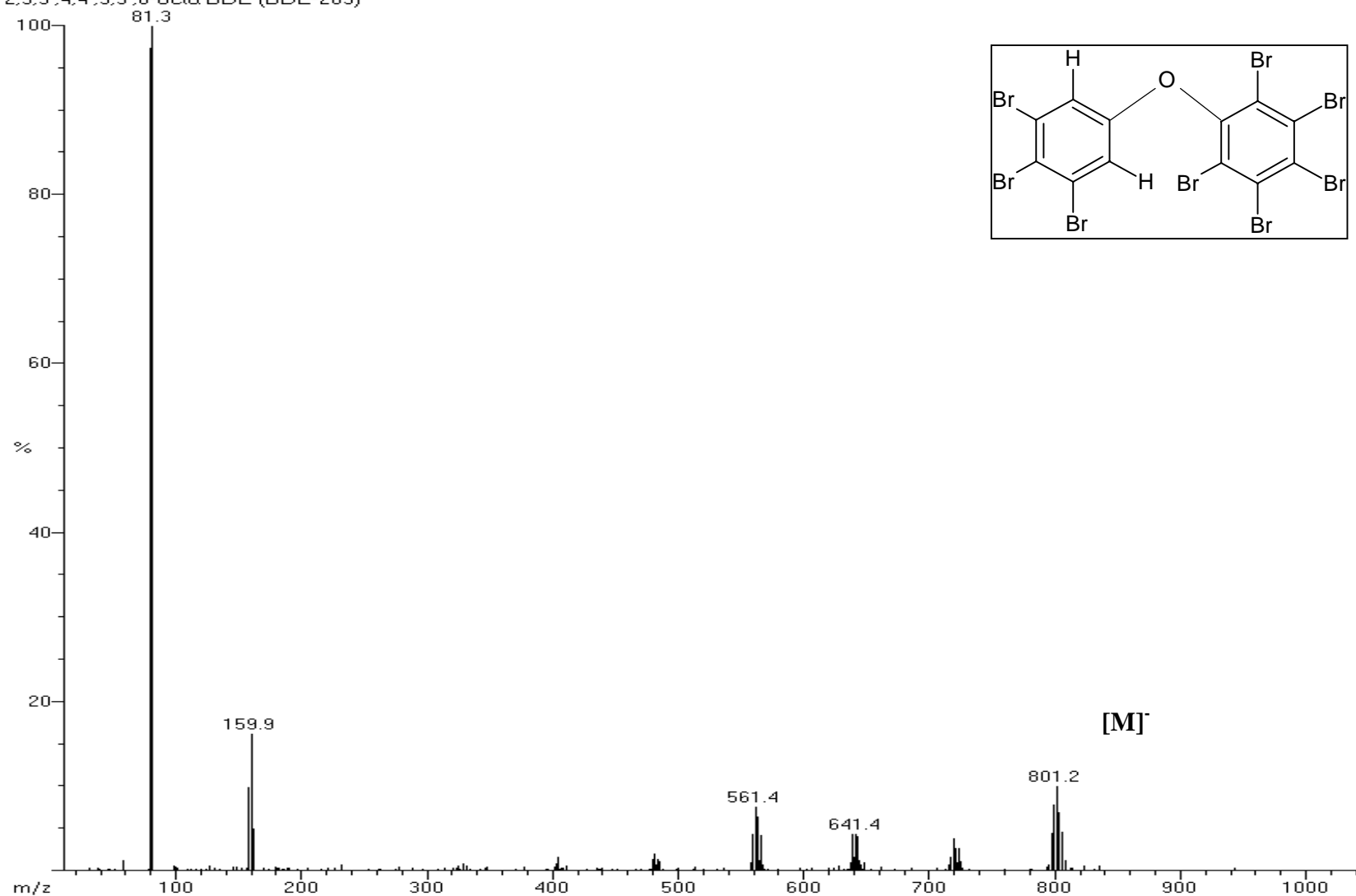
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bde204 | bde204 450pg  
Scan: 683(733-747) TIC=3320596 Base=37%FS #ions=579 RT=18.92  
2,2',3,4,4',5,6,6'-octa BDE



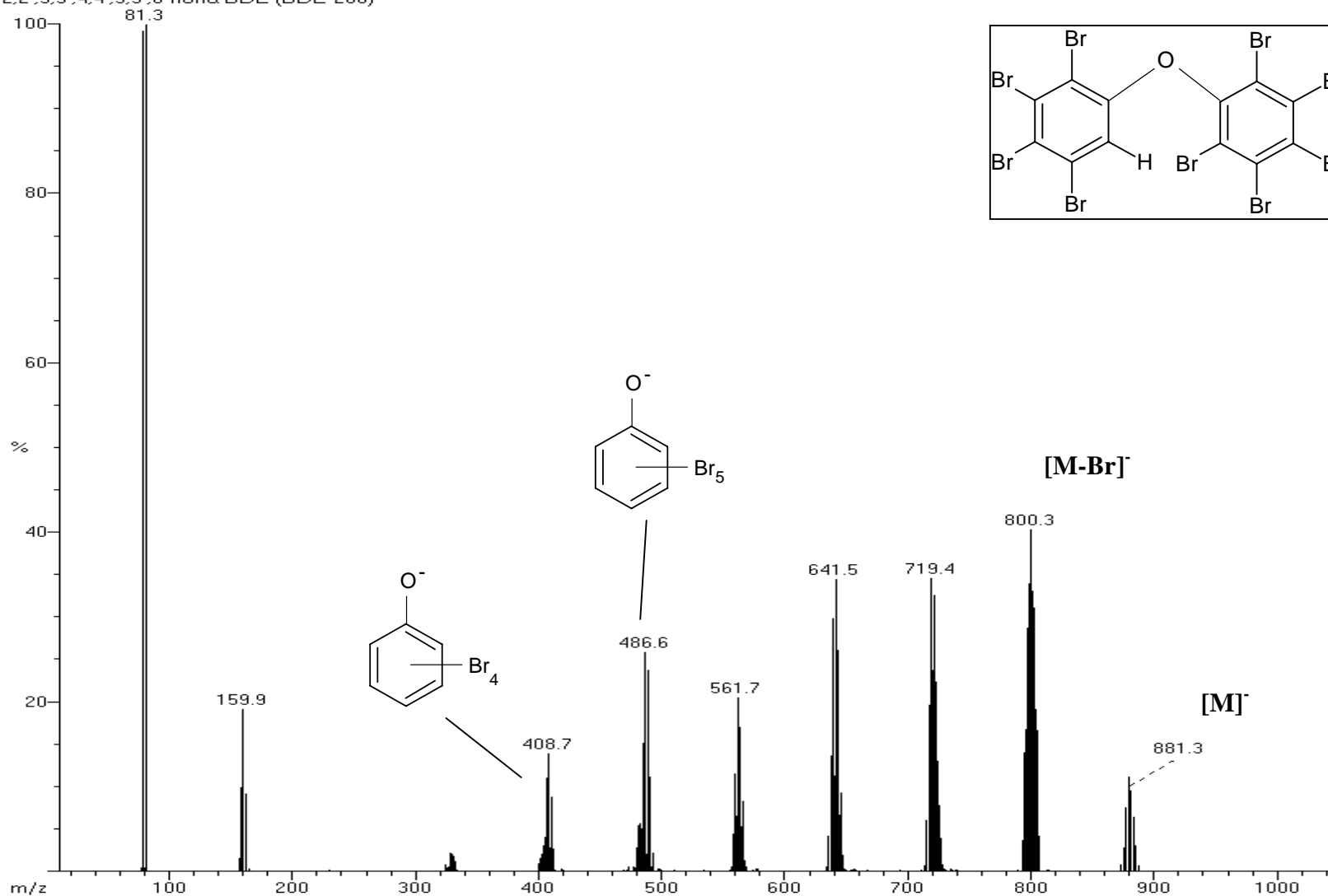
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bde205a | bde205 450pg  
Scan: 720(676-690) TIC=918549 Base=22%FS #ions=406 RT=19.67  
2,3,3',4,4',5,5',6-octa BDE (BDE-205)



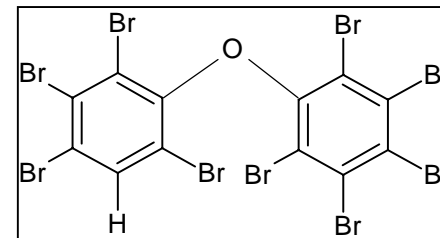
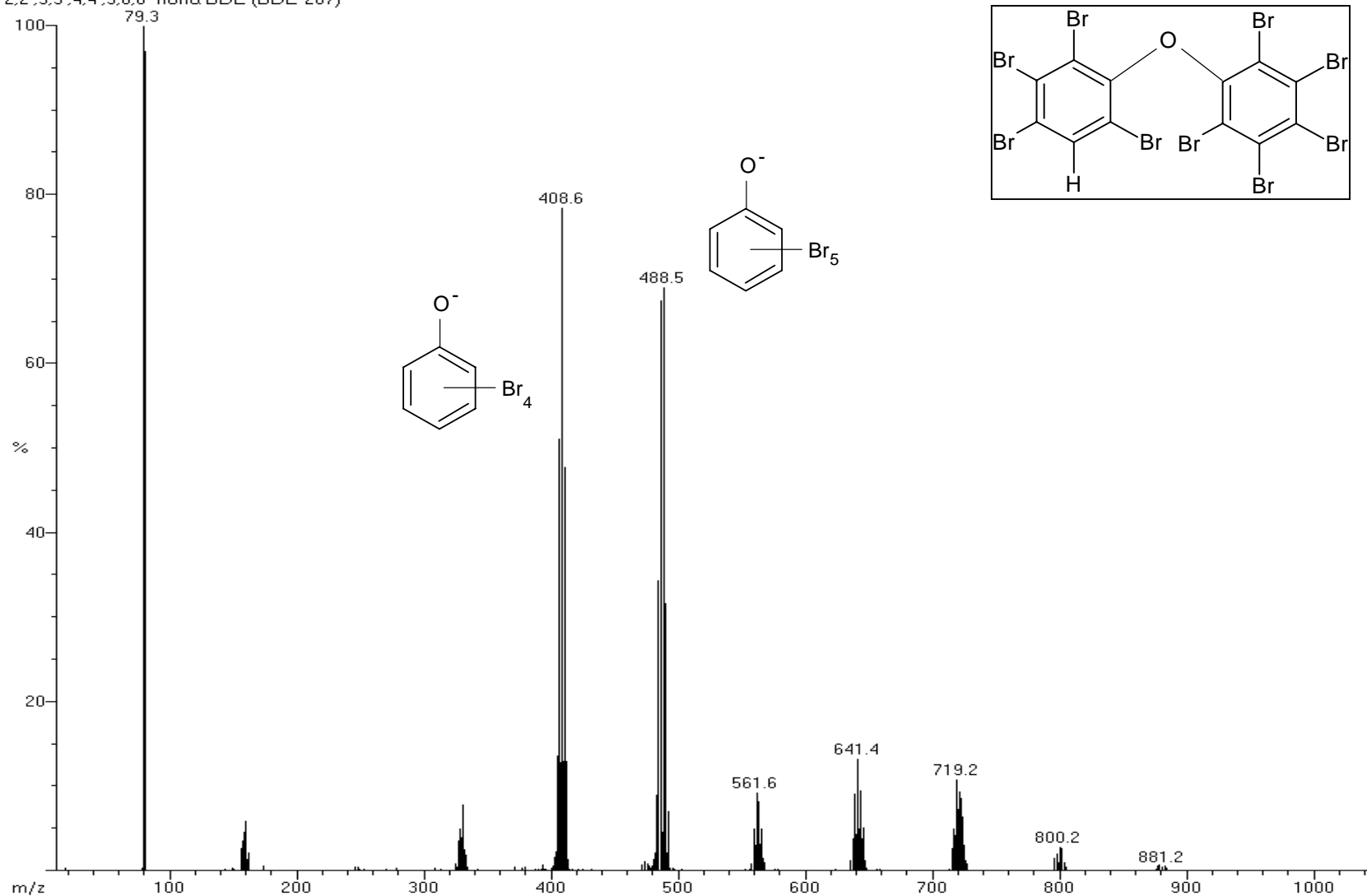
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bdemxe500\_1000\_2500ng.c | bdemxe 500/1000/2500ng @200C  
 Scan: 796(753-767) TIC=9486434 Base=73.5%FS #ions=1651 RT=21.22  
 2,2',3,3',4,4',5,5',6-nona BDE (BDE-206)



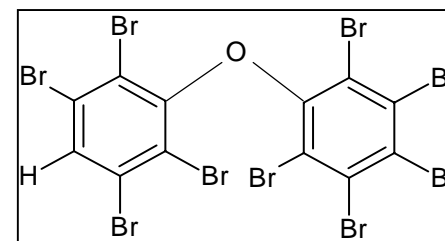
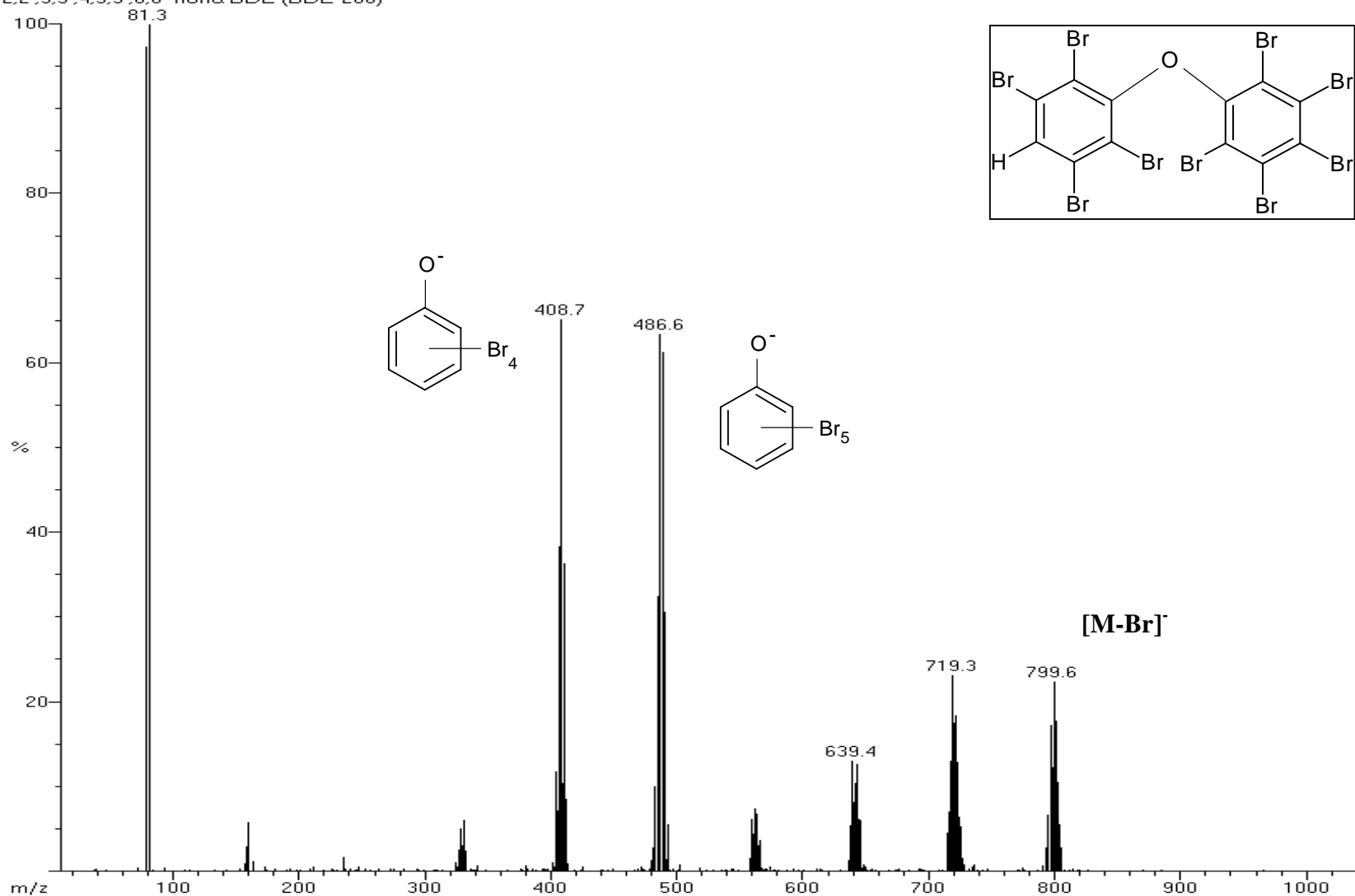
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bde207 | bde207 500pg  
Scan: 780(726-740) TIC=4787000 Base=47.8%FS #ions=723 RT=20.88  
2,2',3,3',4,4',5,6,6'-nona BDE (BDE-207)



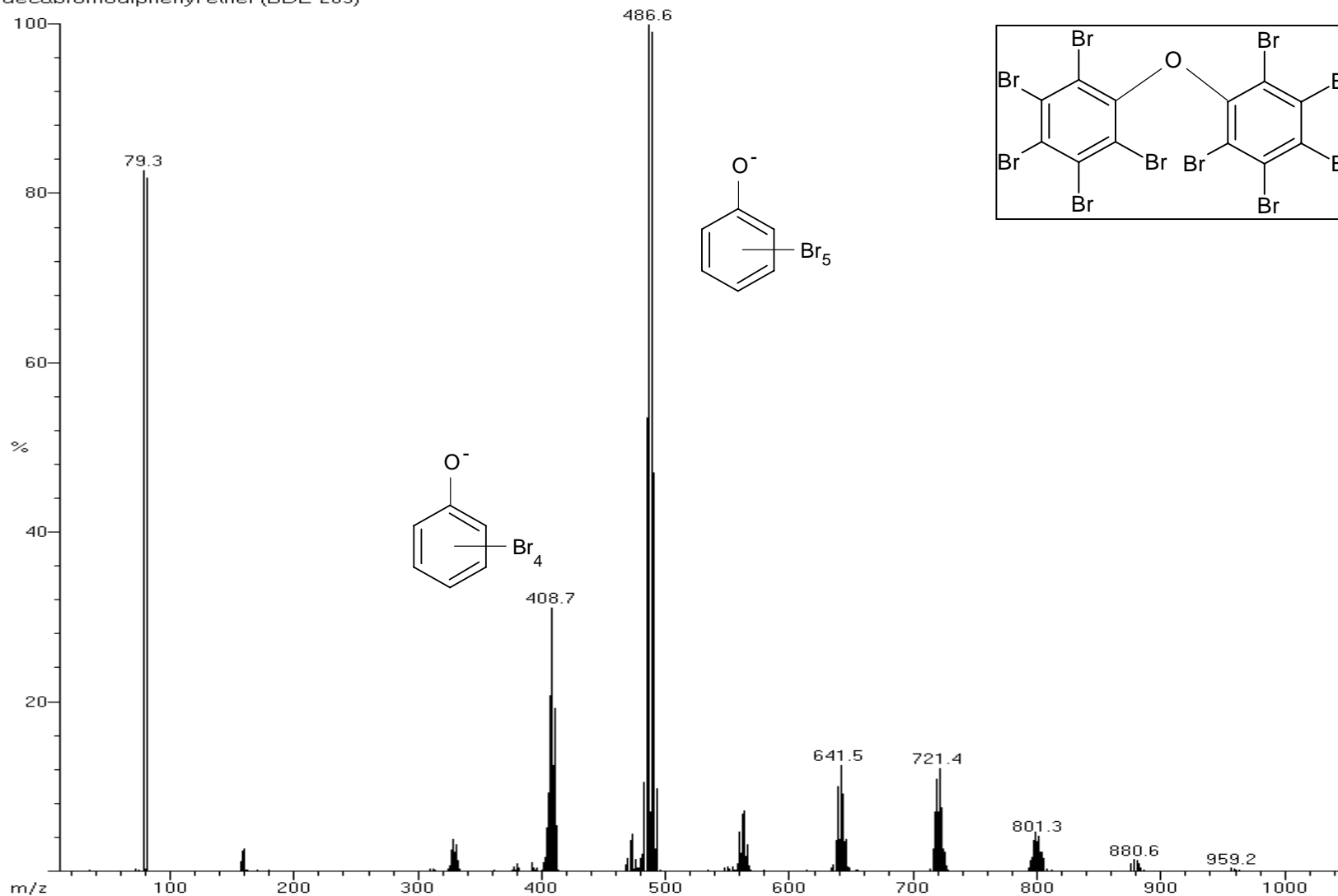
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bde208 | bde208 500pg  
Scan: 773(828-842) TIC=2710574 Base=23.7%FS #ions=563 RT=20.75  
2,2',3,3',4,5,5',6,6'-nona BDE (BDE-208)





bdemxe500\_1000\_2500ng c | bdemxe 500/1000/2500ng @200C  
Scan: 902(962-976) TIC=6712199 Base=70.8%FS #ions=1246 RT=23.38  
decabromodiphenyl ether (BDE-209)



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