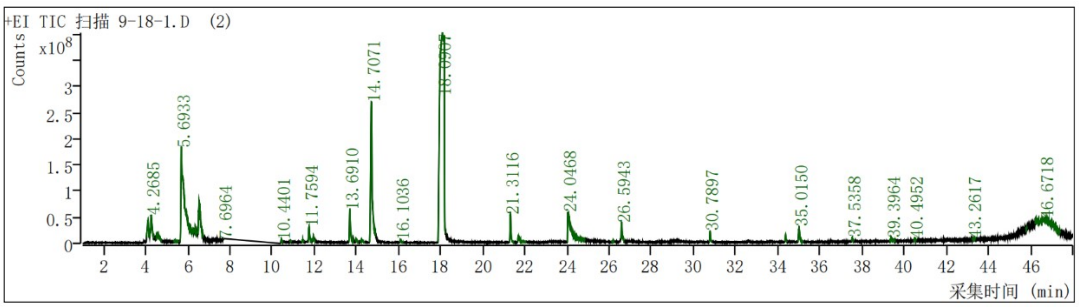
General Procedure 3: Validation experiments

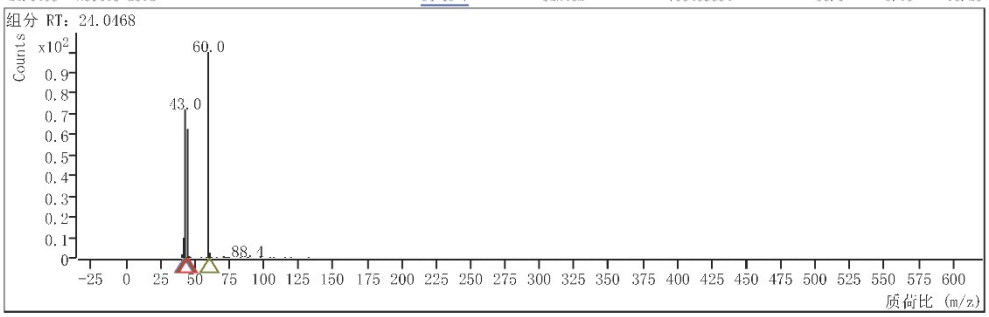
To investigate the rationality of the proposed mechanism, we employed GC-MS to analyze the reaction of *N*-benzyl-*o*-phenylenediamine (**1a**) with acetoin (**2a**). The results indicate that acetic acid may be derived from the oxidation of acetaldehyde during the reaction. Subsequently, we also investigated the reaction of *N*-benzyl-*o*-phenylenediamine (**1a**) with 1-hydroxychlorohexyl phenyl ketone (**2c**), which resulted in the formation of cyclohexanone. These two experiments support our hypothesis regarding the reaction mechanism. Furthermore, in the reaction of *N*-benzyl-*o*-phenylenediamine (**1a**) with ethyl 3-methyl-2-oxobutanoate (**4a**), we detected the formation of ethyl trifluoroacetate, suggesting that ethanol was generated during the process.

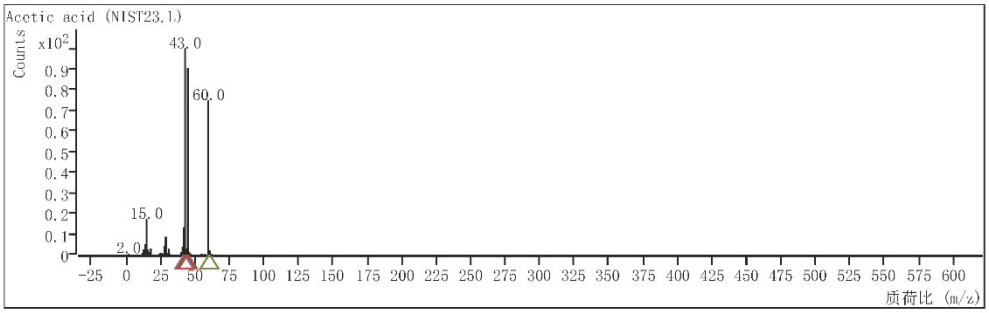
GC–MS analyses were performed using a 7000D GC/TQ instrument (Aglient). The reaction mixture were analyzed on a DB-FFAP column (60 m x 250 μm x 0.25 μm). Helium at a constant flow rate of 1.2 mL/min was used as carrier gas. The injector was held at 250°C. The temperature rise program of the gas chromatography parameter column incubator was as follows: the oven temperature was held at 50°C for 2 min, then programmed to 80°C for 2 min, and finally kept at 230°C for 3 min. The mass spectrum parameters were set as follows: EI source, ionization energy 70 eV, ion source temperature 230◦C, and scanning range m/z 40–550. Compound analysis was carried out through the NIST database.

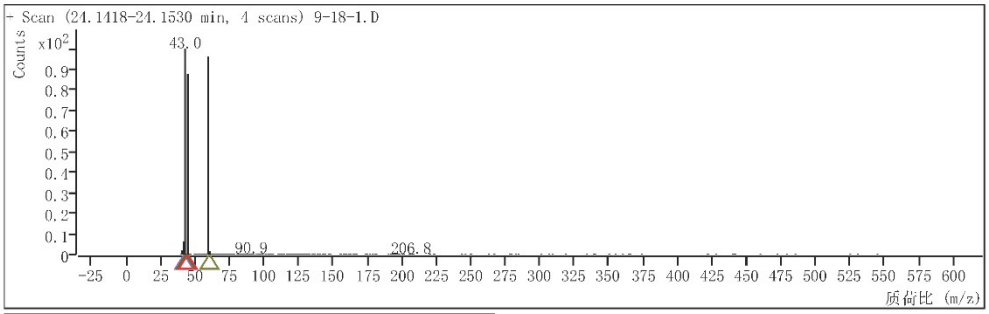


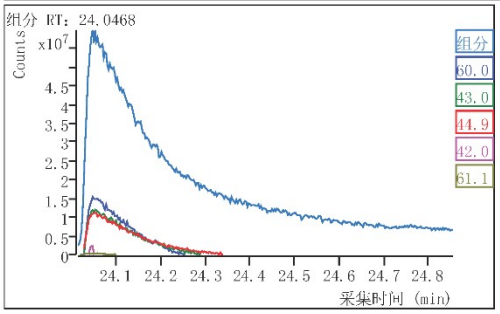
未知物分析报告 - 最佳匹配

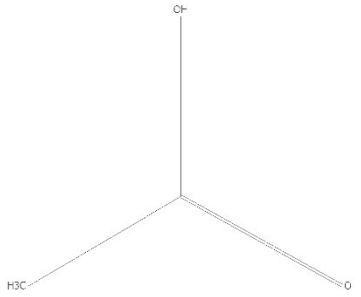
RT	化合物名称	CAS#	分子式	面积 MI	匹配分数	面积 (%)_T	面积 (%)_M
24.0468	Acetic acid	64-19-7	C2H4O2	768459884	88.0	6.13	16.23

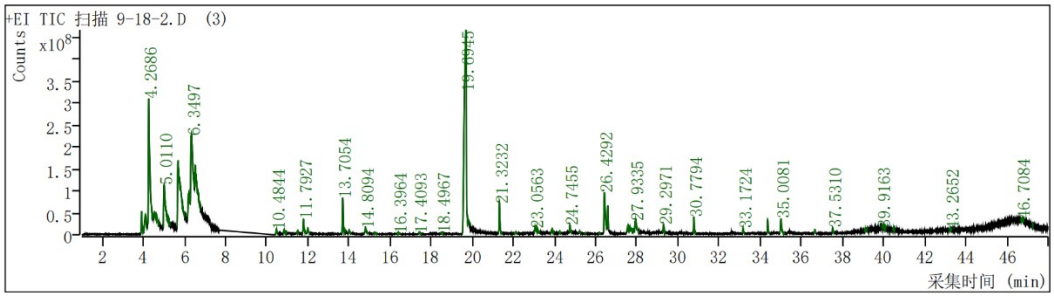








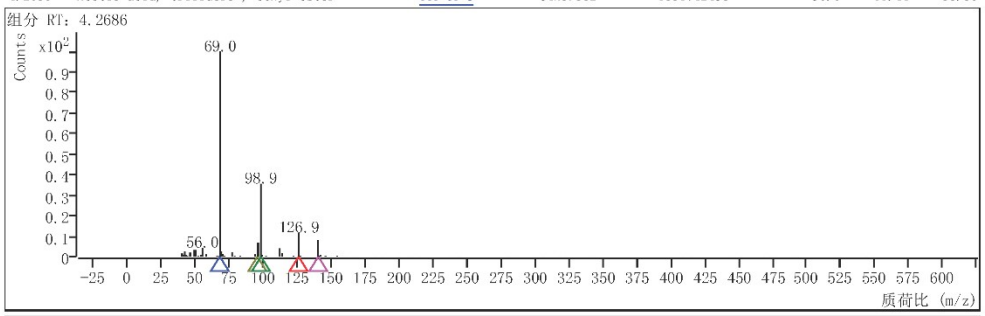


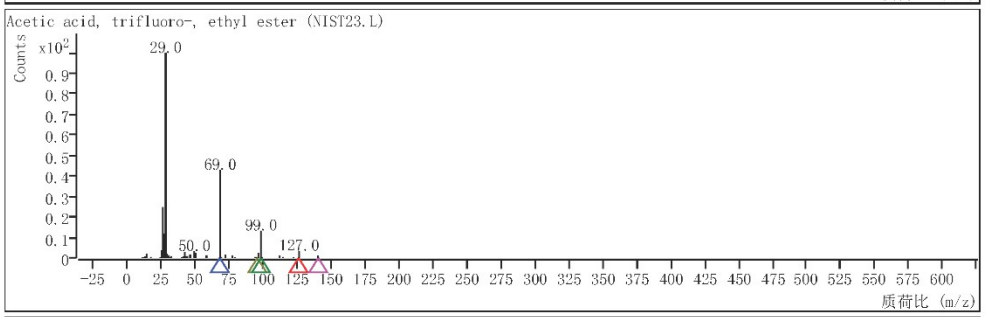


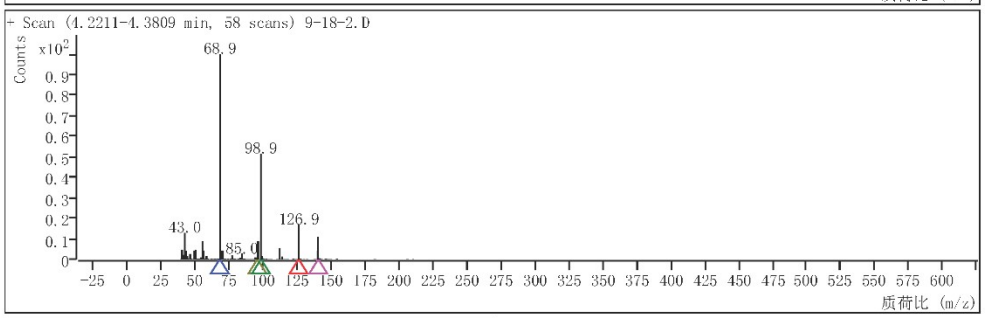
未知物分析报告 - 最佳匹配

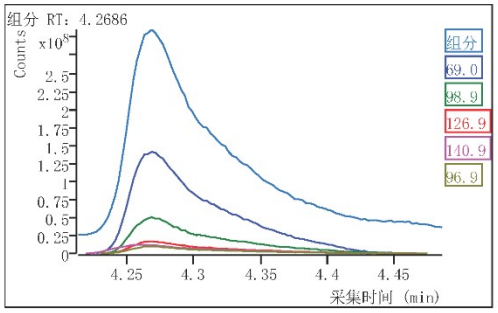
Agilent | Technologies

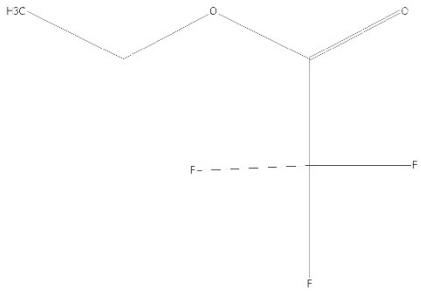
RT	化合物名称	CAS#	分子式	面积 MI	匹配分数	面积 (%)_T	面积 (%)_M
4.2686	Acetic acid, trifluoro-, ethyl ester	383-63-1	C4H5F3O2	1381742495	90.0	11.44	38.30











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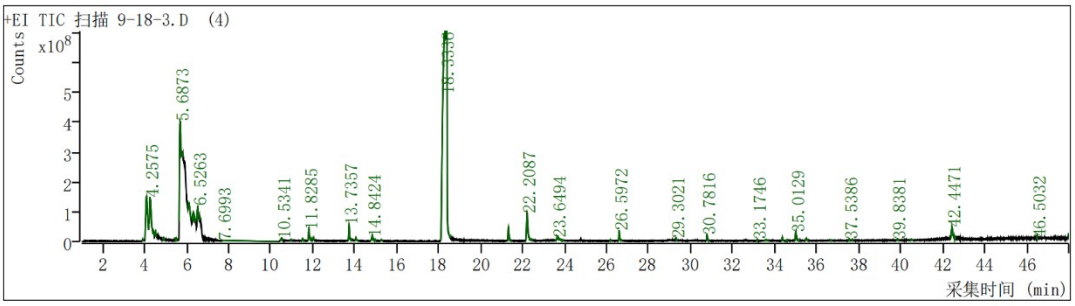

RT	化合物名称	CAS#	分子式	面积 MI	匹配分数	面积 (%)_T	面积 (%)_M
18.3336	Cyclohexanone	108-94-1	C6H10O	7077993128	92.1	44.36	100.00