Electronic Supplementary Material (ESI) for RSC Advances.

SUPPLEMENTARY MATERIALS

Metal- and oxidant-free electrochemically promoted oxidative coupling of amines

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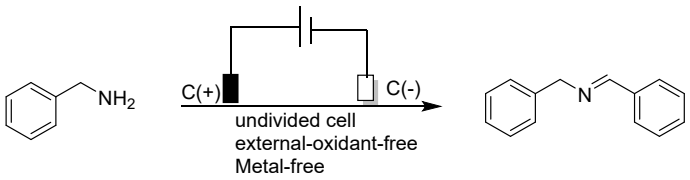
1. General information:

All reagents and materials (purchased from Aladdin) were used as received, solvents of analytical grade were used for the reaction without further purification.

Characterization and instruments. The GC analyses were performed on Shimadzu GC-2014C with an FID detector equipped with a Cap Hp-5 Sil capillary column. The GC mass spectra (GC-MS) were recorded on Agilent 7890B-7000D. The instrument for electrolysis is ElectraSyn 2.0 (made in America), the Carbon plate (53 mm * 8 mm * 1.5 mm) and the Platinum plate (54 mm * 8 mm * 1.8 mm) was purchased from Aika (Guangzhou) instrument equipment Co., LTD.

General electrocatalysis procedure for oxidation of amines to imines



Under 5 V constant voltage conditions, a dried ElectraSyn 2.0 vial equipped with a stir bar was loaded with benzylamine (0.25 mmol), TBEA (7 mg,) in CH_3CN (3.0 mL) was stirred at 25 ℃. The tube was equipped with carbon plate (53 mm * 8 mm * 1.5 mm) as the anode and cathode. The reaction mixture was stirred and electrolyzed at a constant voltage of 5 V under room temperature for 10 h. After the reaction was completed, the resulting mixture was finally analyzed by GC-MS and GC.

Furthermore, a radical scavenger 4 equiv. 2,2,6,6-tetramethylpiperidinooxy (TEMPO) was added to the electrolysis of benzylamine under the standard conditions. After electrolysis, the reaction mixture samples were analyzed by GC-MS. The intermediate was detected by GC-MS analysis (Figure S2). GC-MS results showed that benzaldehyde might be involved in the electrochemical conversion.

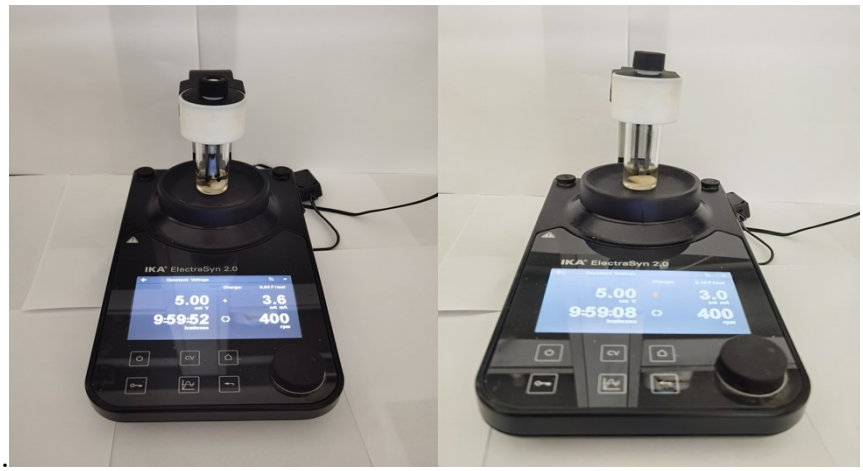


Figure S1. Set-up of experiments (the photographs come from our laboratory).

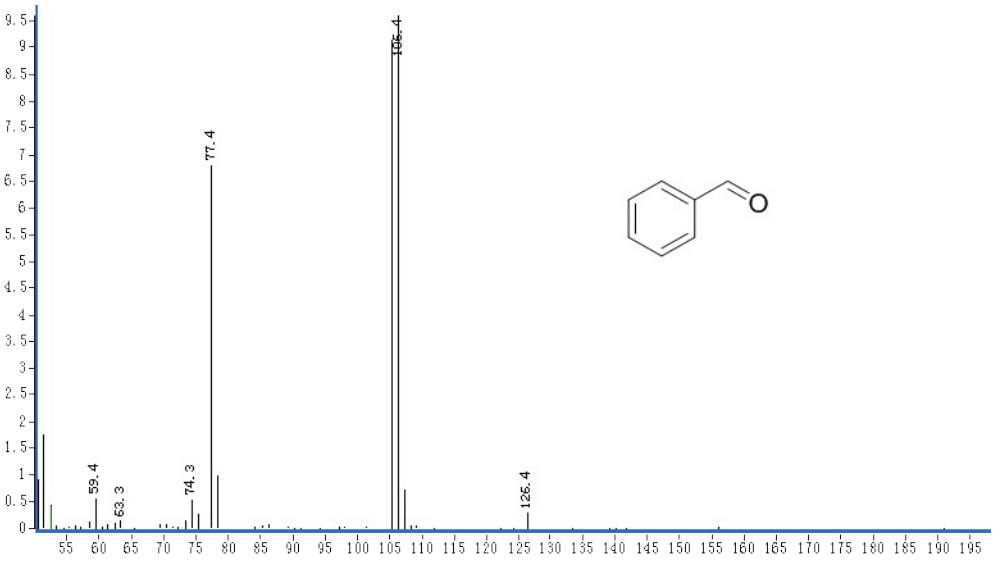


Figure S2. GC-MS analysis of the intermediate benzaldehyde

2. The GC analysis and MS spectrum of products

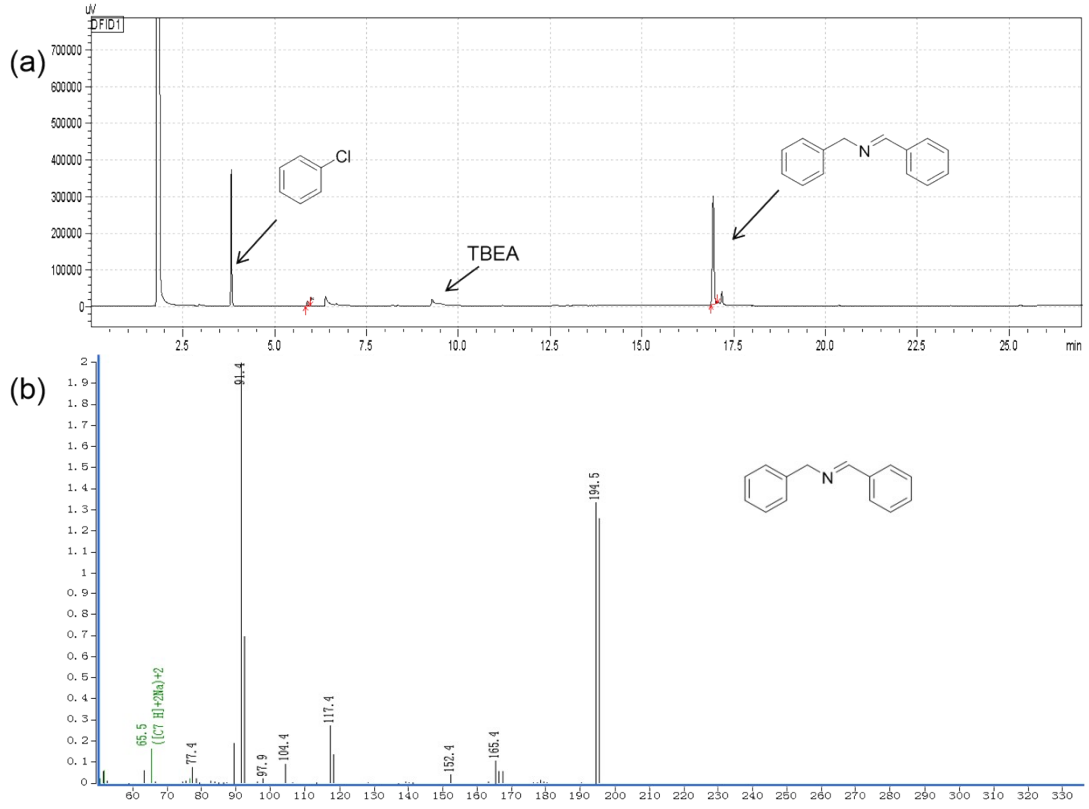


Figure S3. (a) The GC analysis of product (Table 2, entry 1); (b) The MS spectrum of product (Table 2, entry 1).

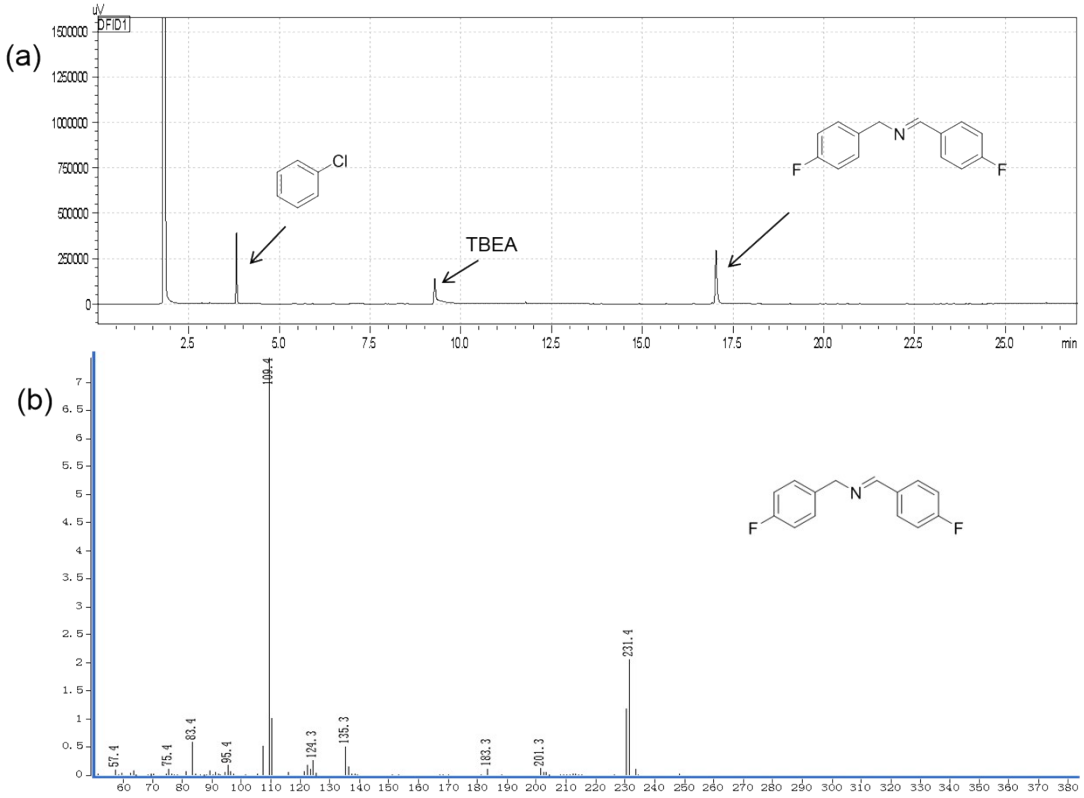


Figure S4. (a) The GC analysis of product (Table 2, entry 2); (b) The MS spectrum of product (Table 2, entry 2).

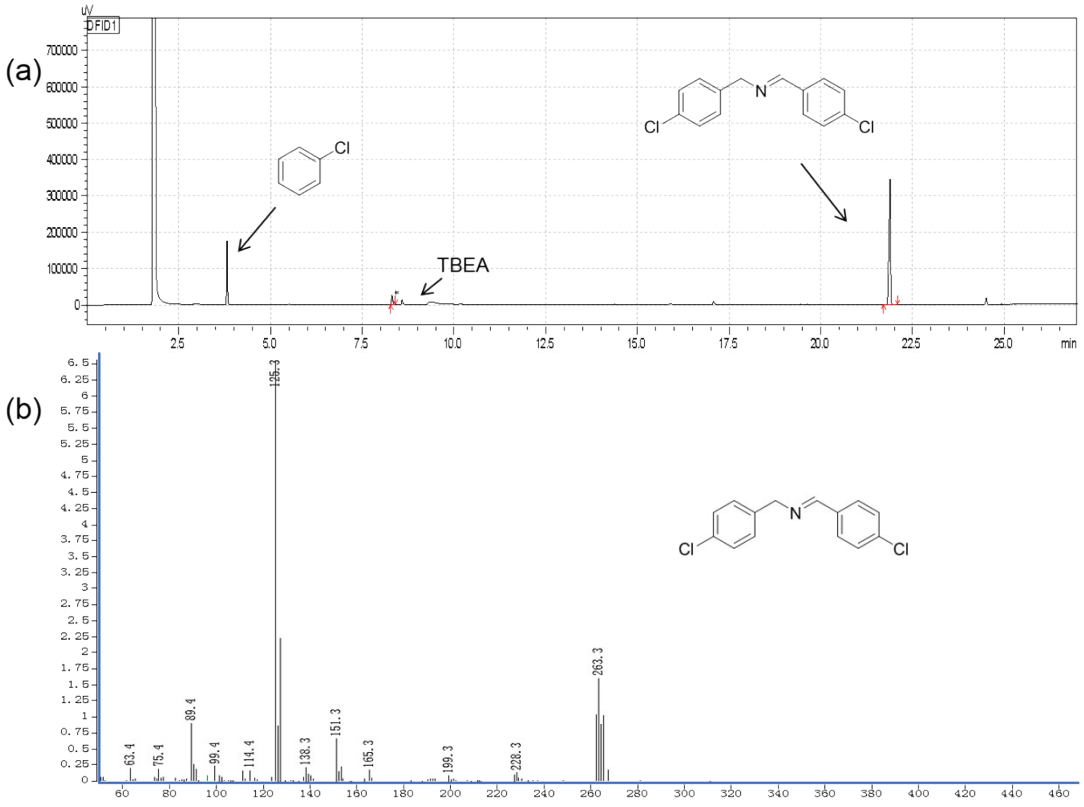


Figure S5. (a) The GC analysis of product (Table 2, entry 3); (b) The MS spectrum of product (Table 2, entry 3).

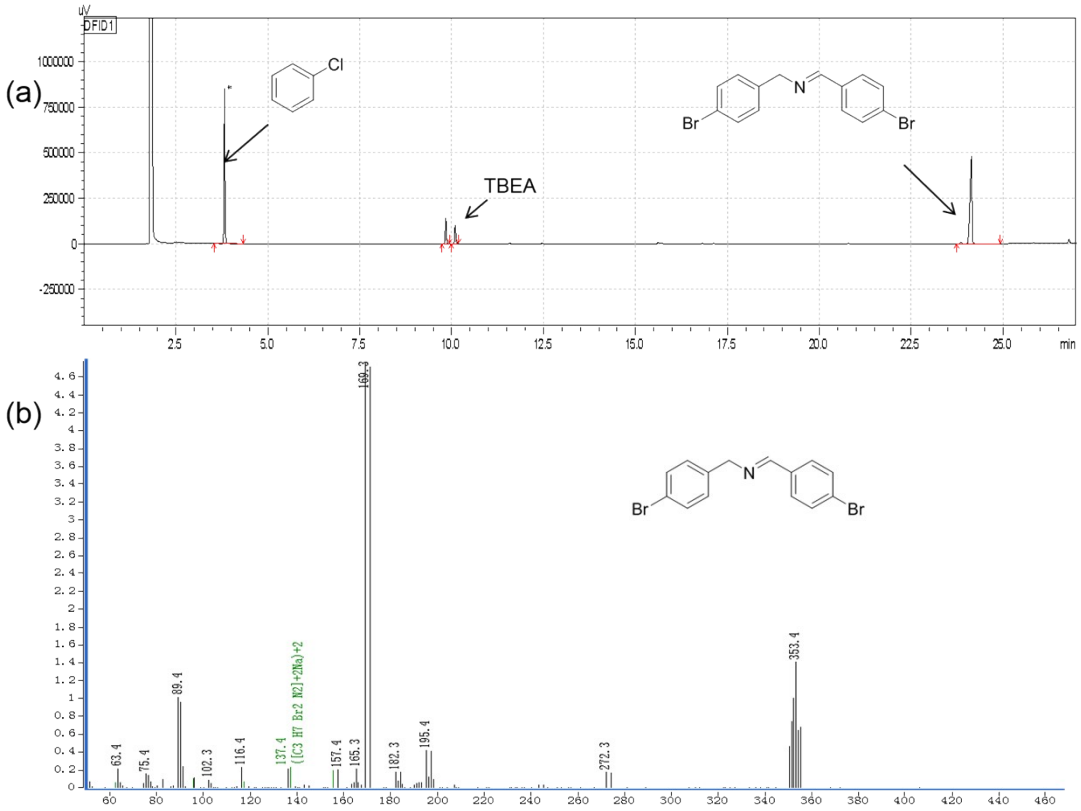


Figure S6. (a) The GC analysis of product (Table 2, entry 4); (b) The MS spectrum of product (Table 2, entry 4).

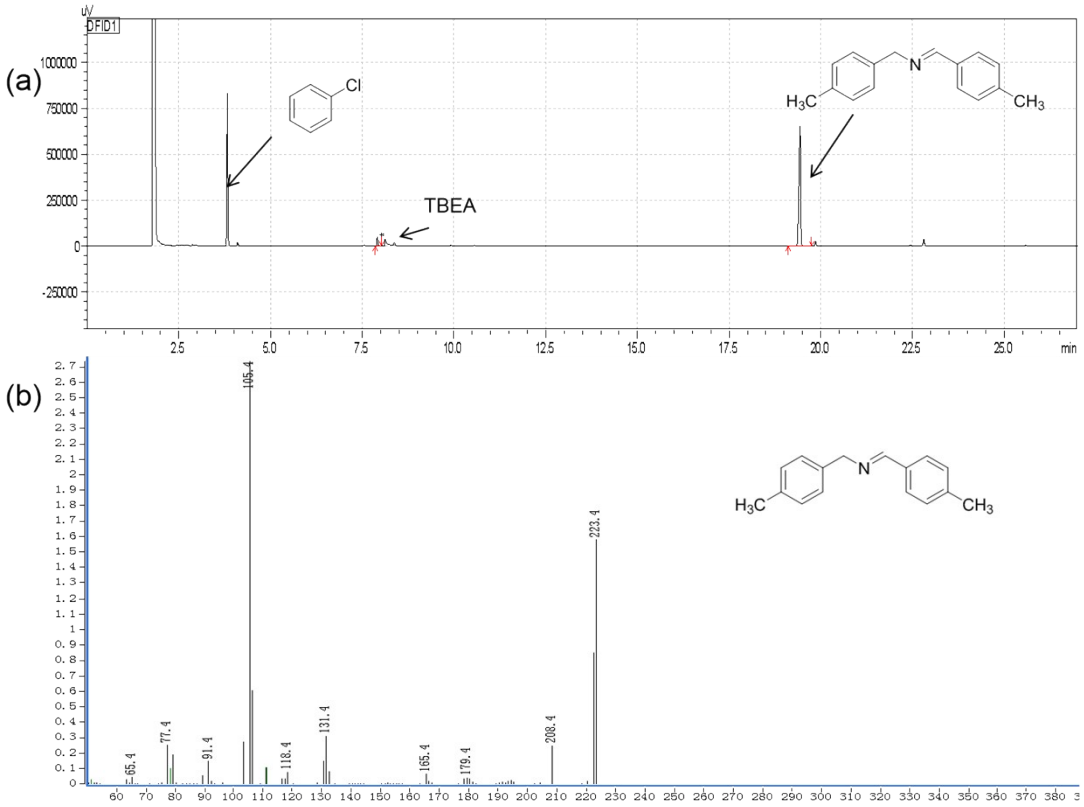


Figure S7. (a) The GC analysis of product (Table 2, entry 5); (b) The MS spectrum of product (Table 2, entry 5).

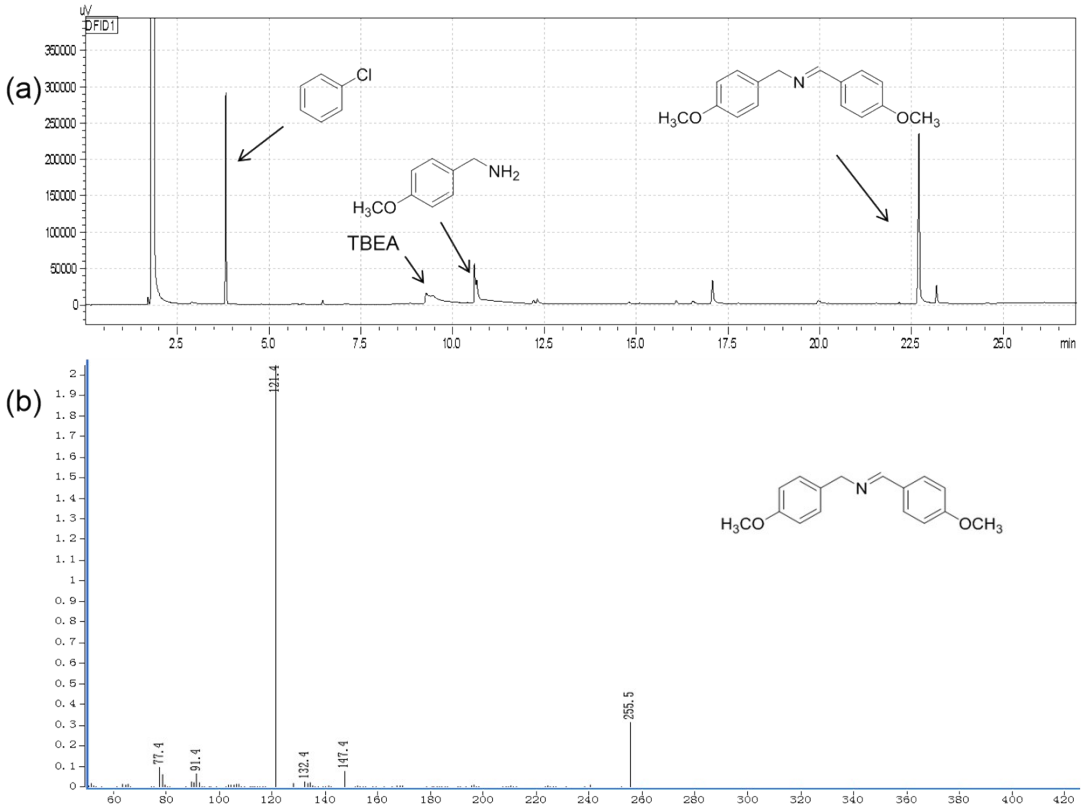


Figure S8. (a) The GC analysis of product (Table 2, entry 6); (b) The MS spectrum of product (Table 2, entry 6).

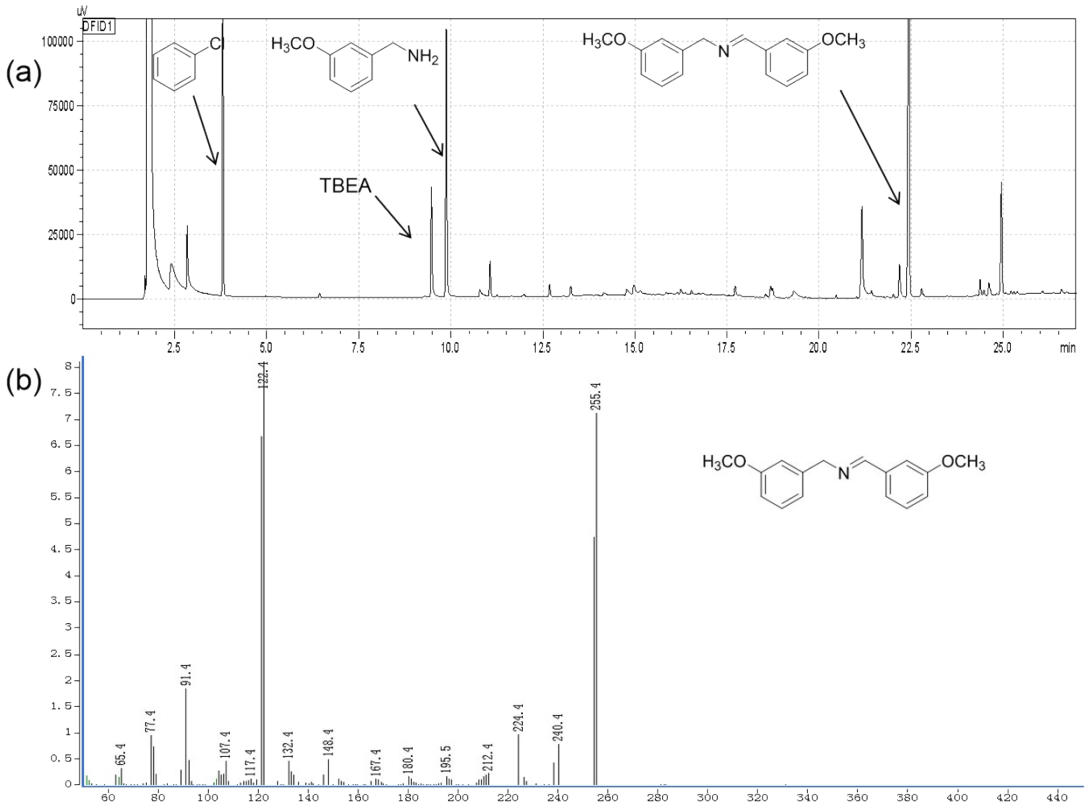


Figure S9. (a) The GC analysis of product (Table 2, entry 7); (b) The MS spectrum of product (Table 2, entry 7).

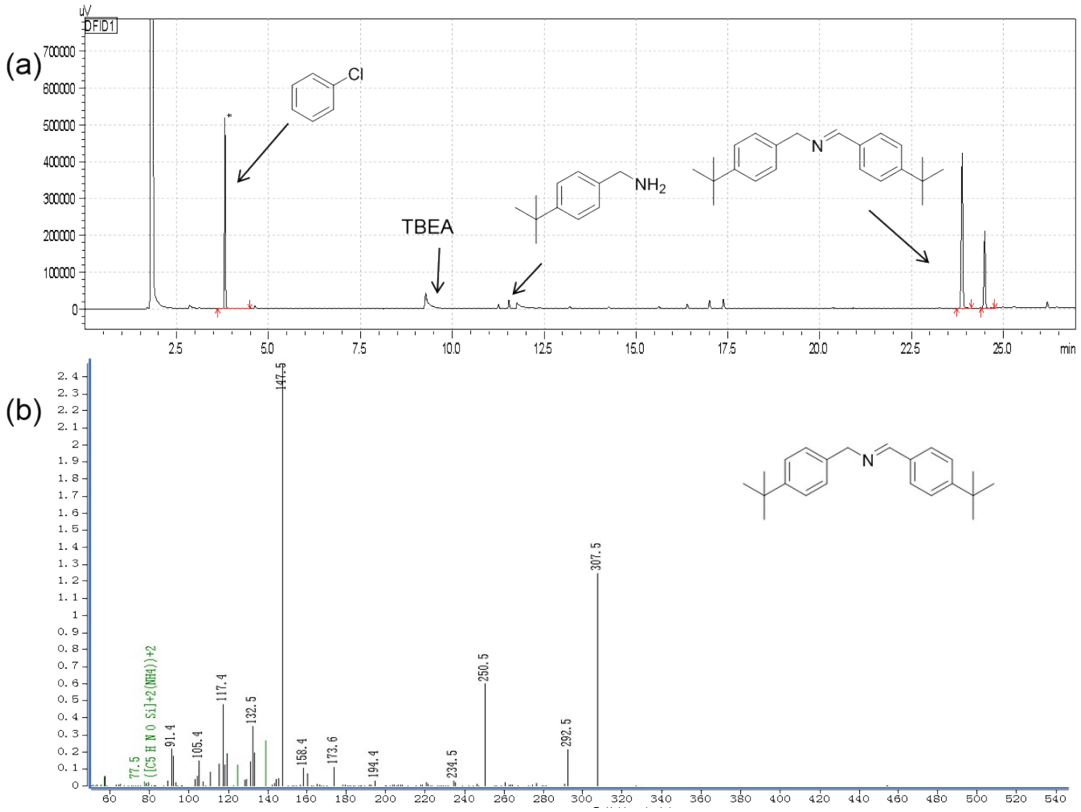


Figure S10. (a) The GC analysis of product (Table 2, entry 8); (b) The MS spectrum of product (Table 2, entry 8).

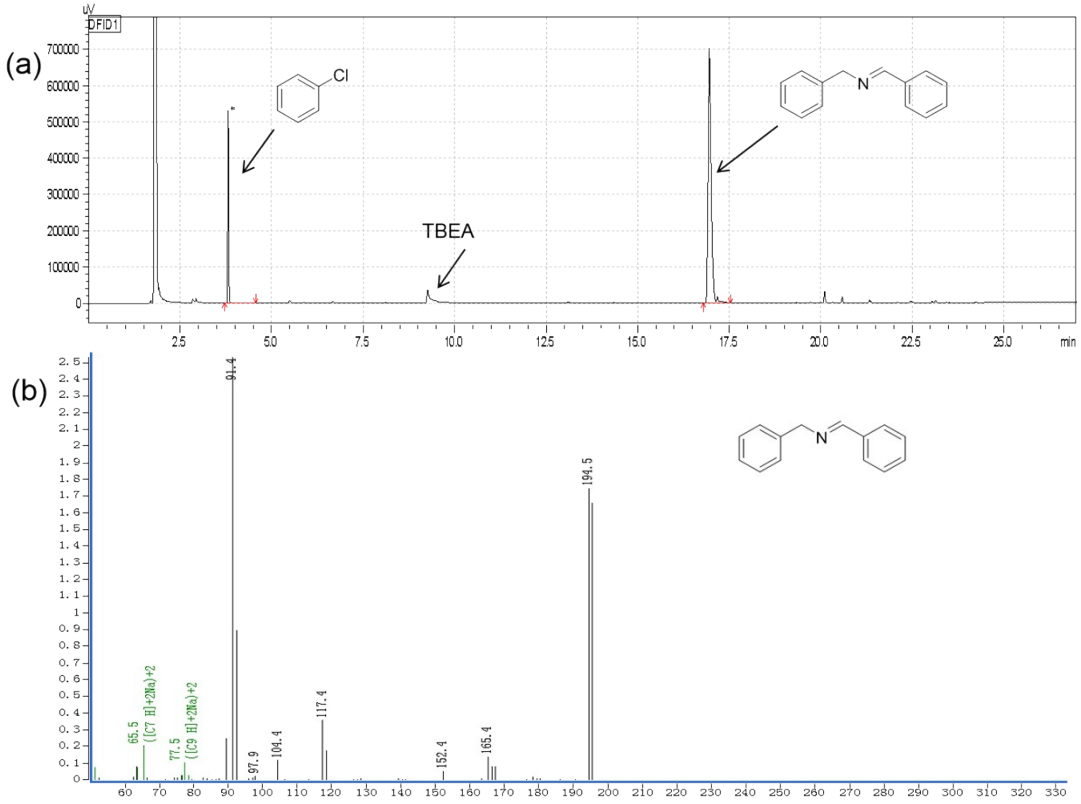


Figure S11. (a) The GC analysis of product (Table 2, entry 9); (b) The MS spectrum of product (Table 2, entry 9).

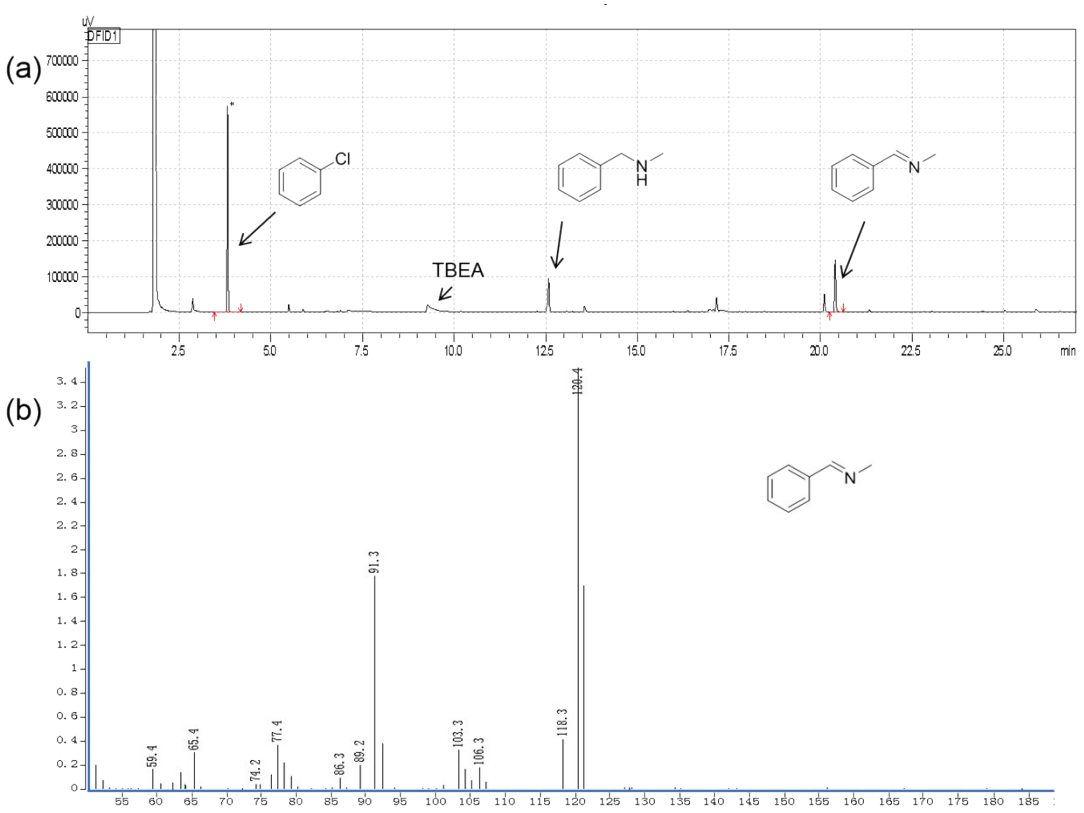


Figure S12. (a) The GC analysis of product (Table 2, entry 10); (b) The MS spectrum of product (Table 2, entry 10).

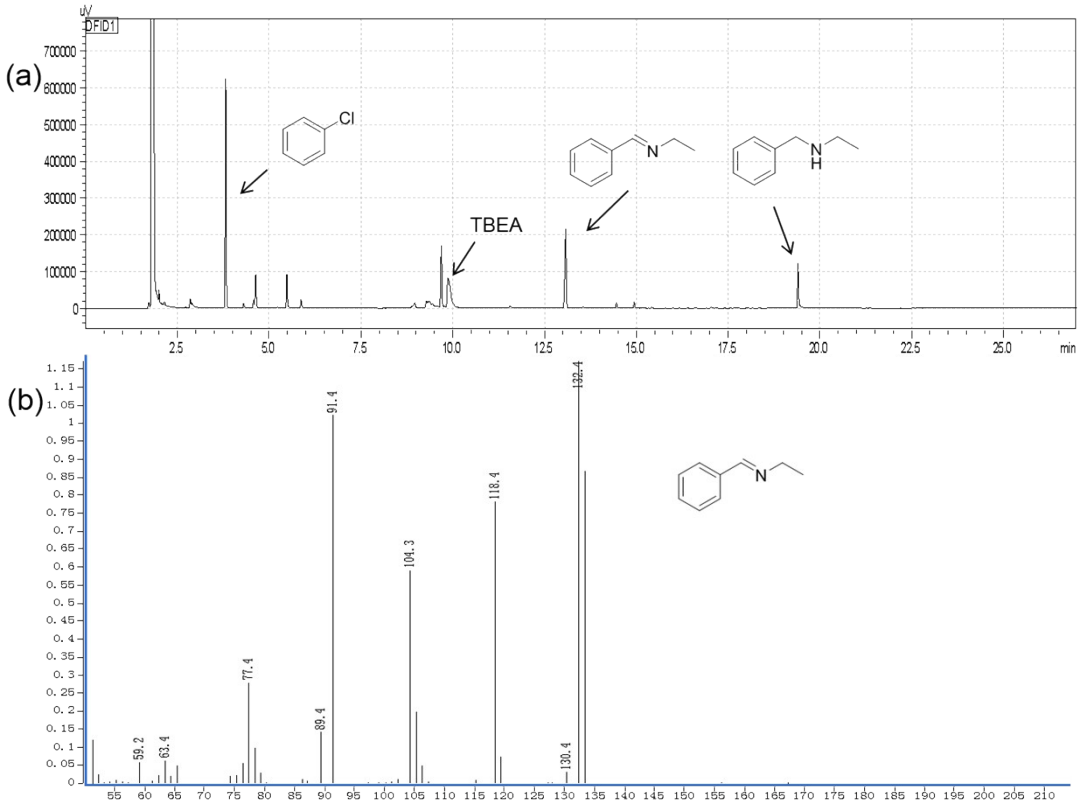


Figure S13. (a) The GC analysis of product (Table 2, entry 11); (b) The MS spectrum of product (Table 2, entry 11).

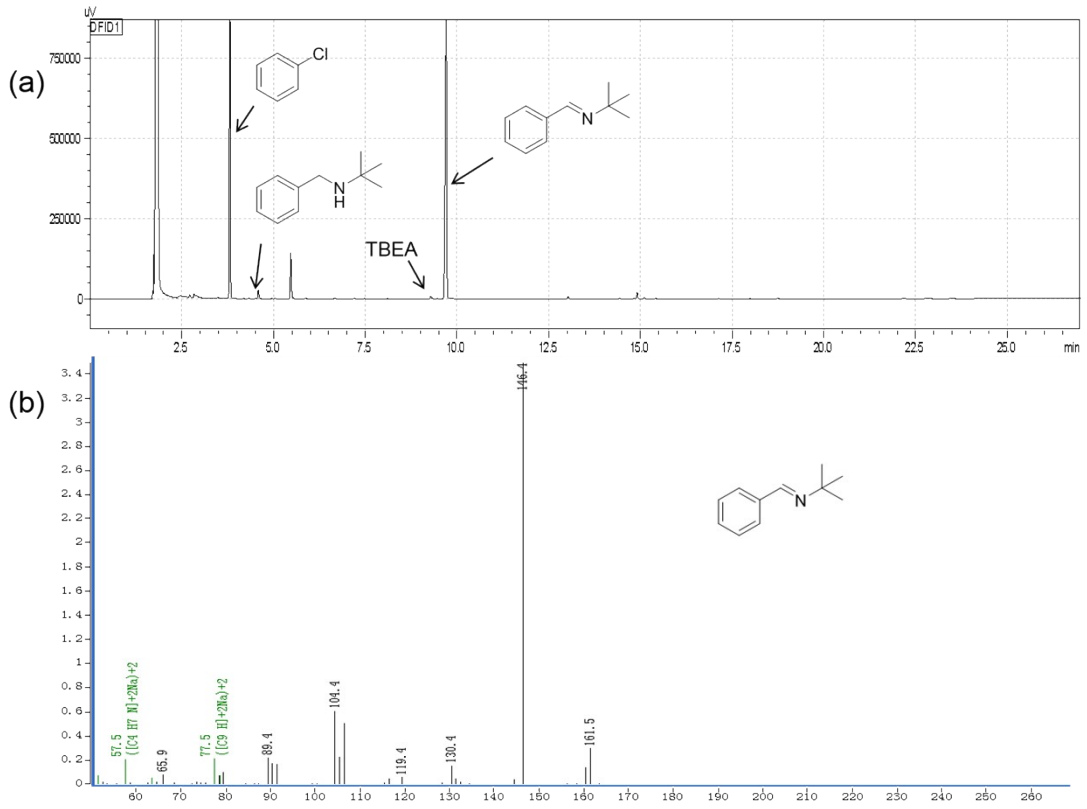


Figure S14. (a) The GC analysis of product (Table 2, entry 12); (b) The MS spectrum of product (Table 2, entry 12).

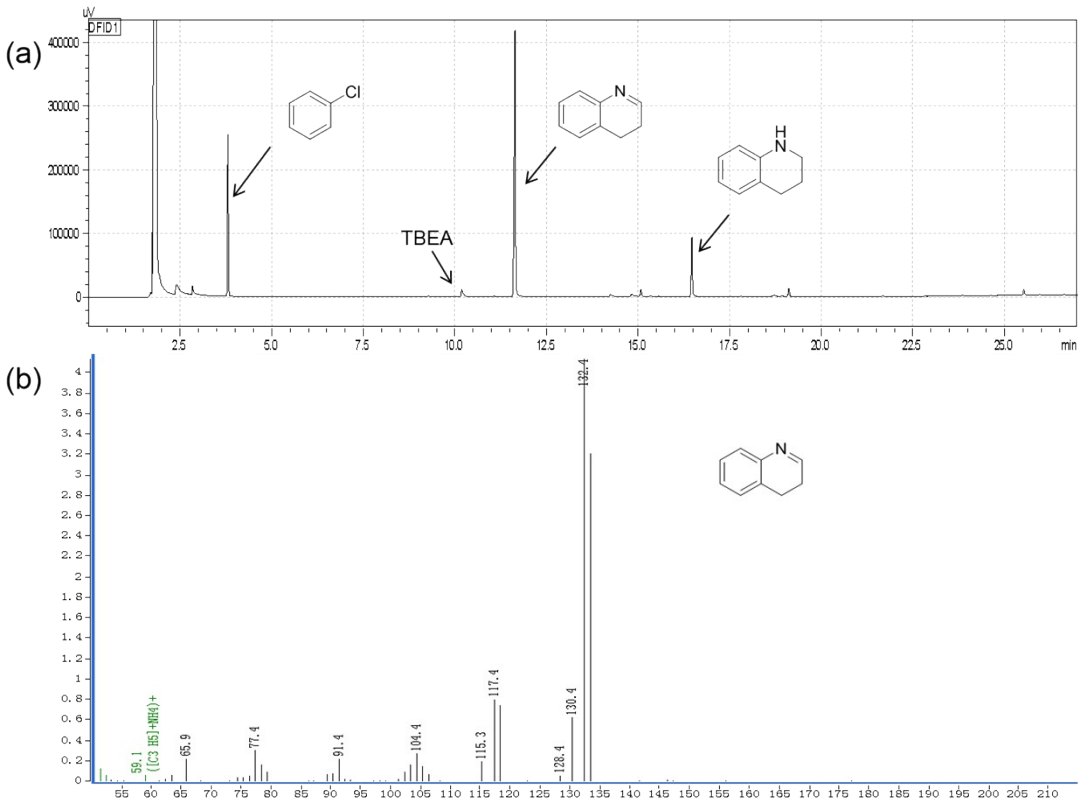


Figure S15. (a) The GC analysis of product (Table 2, entry 13); (b) The MS spectrum of product (Table 2, entry 13).

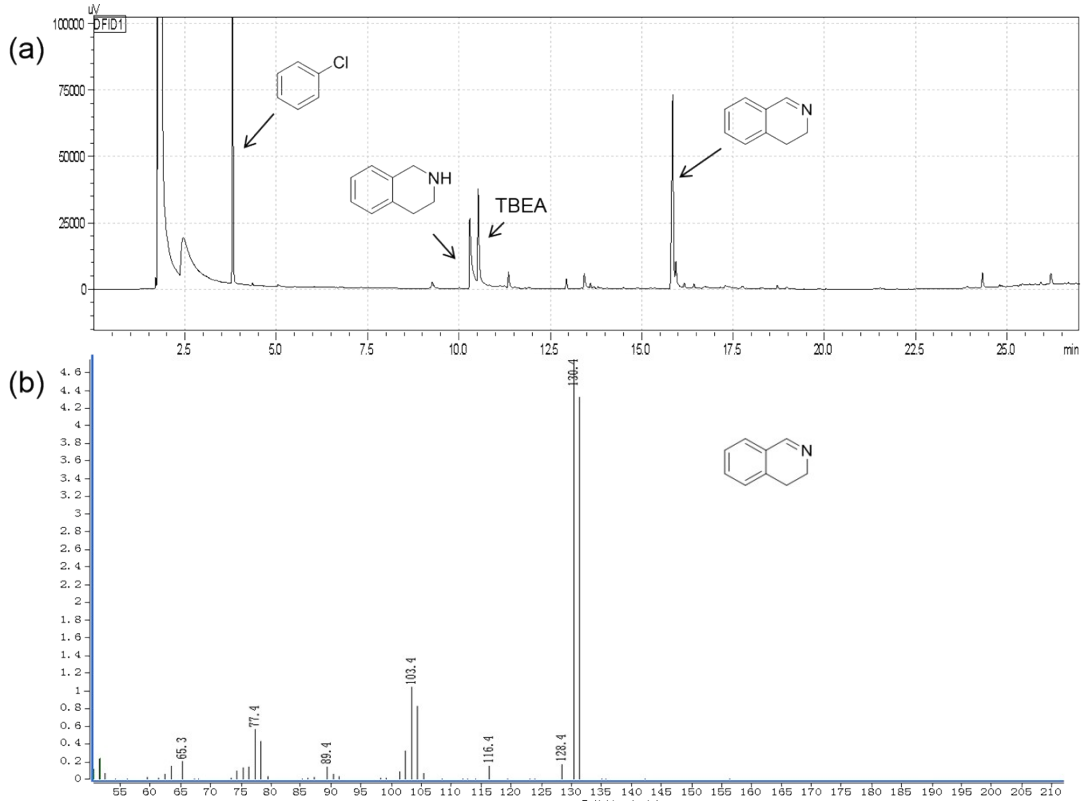


Figure S16. (a) The GC analysis of product (Table 2, entry 14); (b) The MS spectrum of product (Table 2, entry 14).

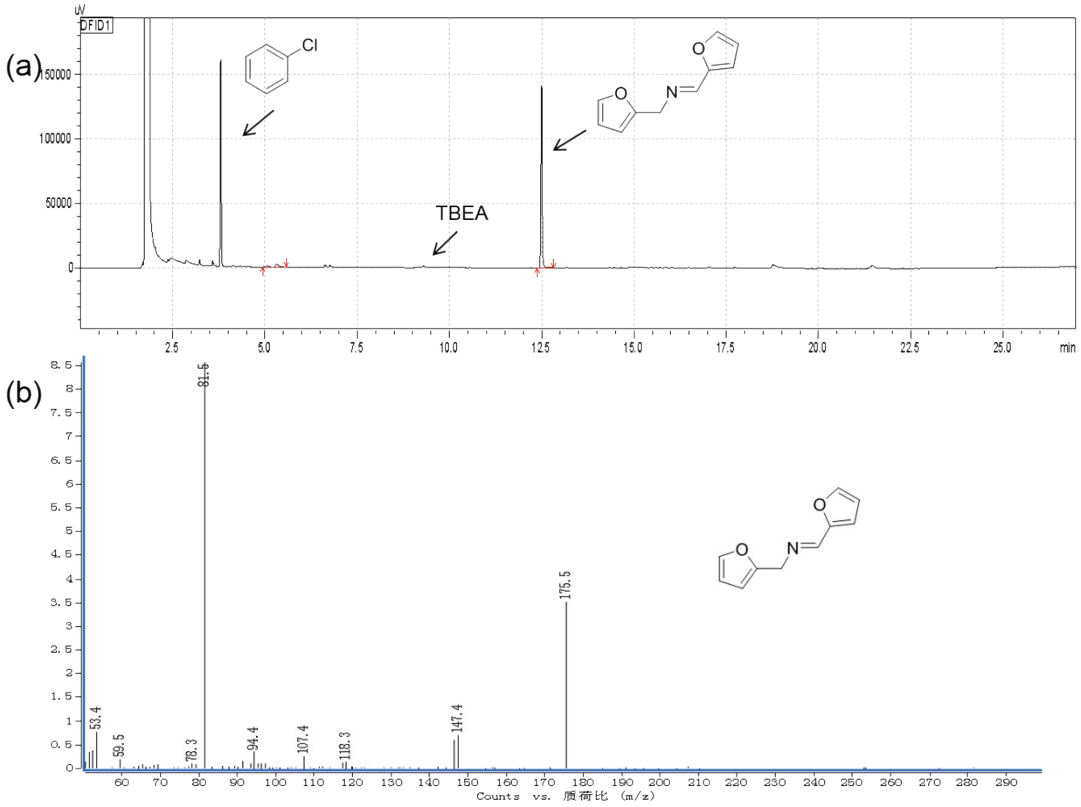


Figure S17. (a) The GC analysis of product (Table 2, entry 14); (b) The MS spectrum of product (Table 2, entry 17).

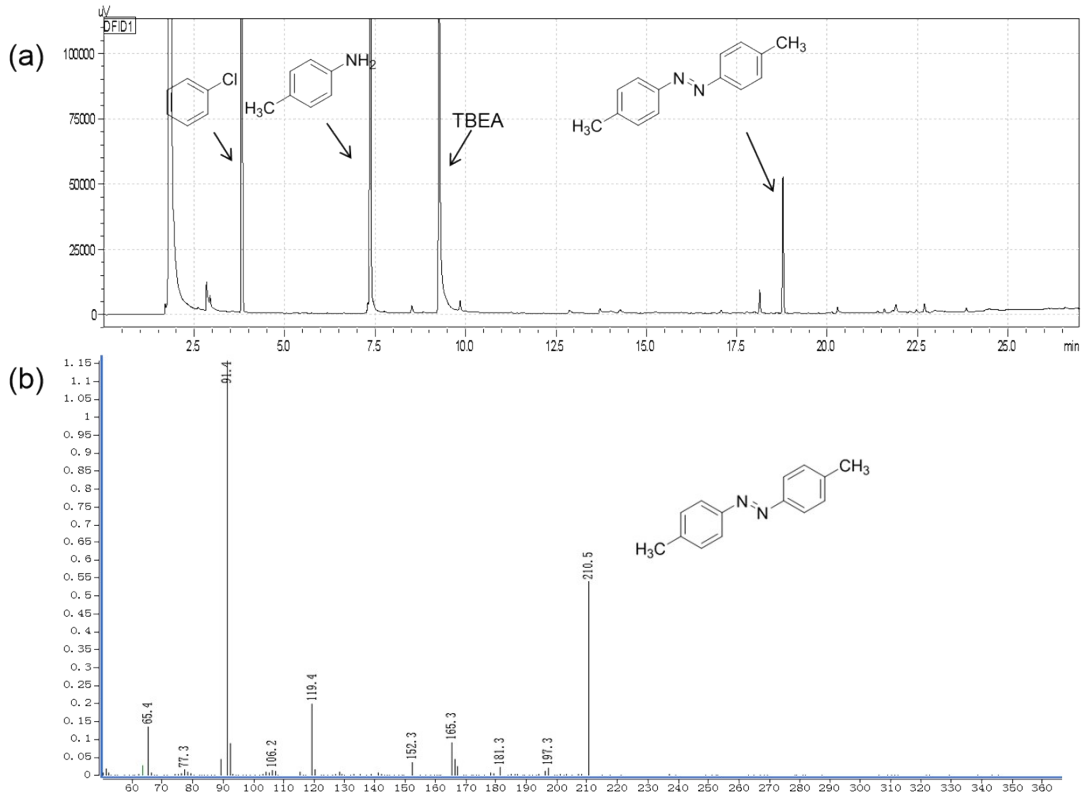


Figure S18. (a) The GC analysis of product (Table 3, entry 1); (b) The MS spectrum of product (Table 3, entry 1).

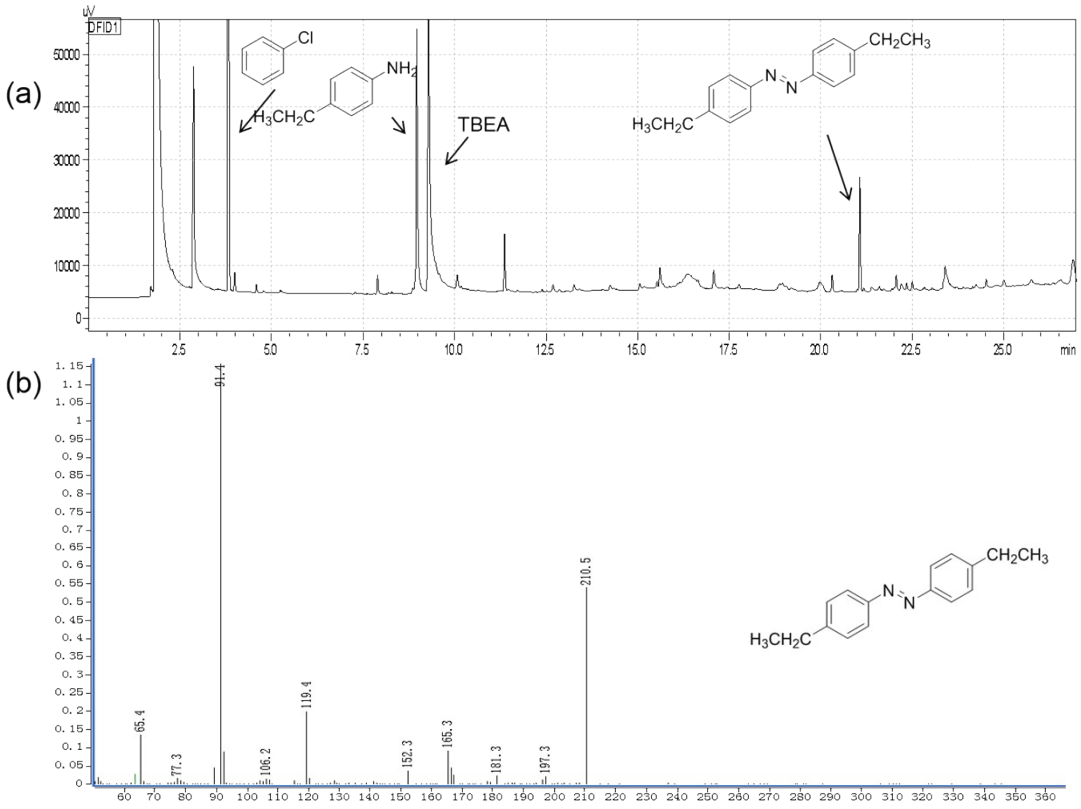


Figure S19. (a) The GC analysis of product (Table 3, entry 2); (b) The MS spectrum of product (Table 3, entry 2).

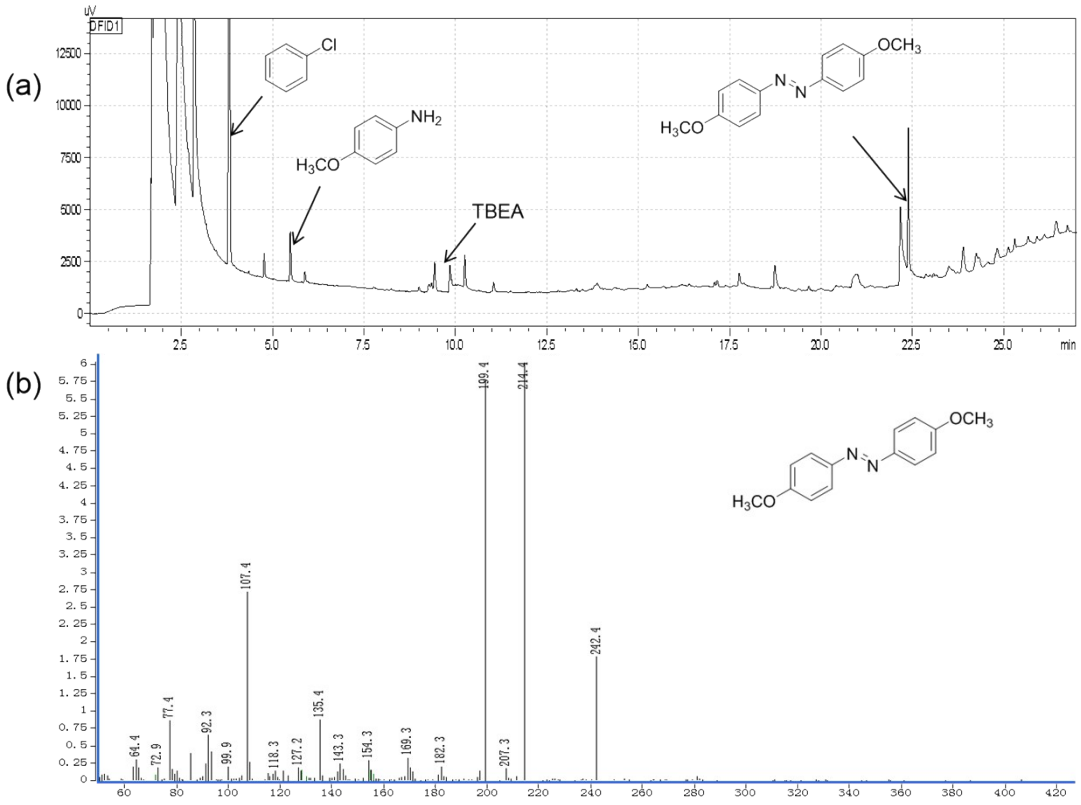


Figure S20. (a) The GC analysis of product (Table 3, entry 3); (b) The MS spectrum of product (Table 3, entry 3).

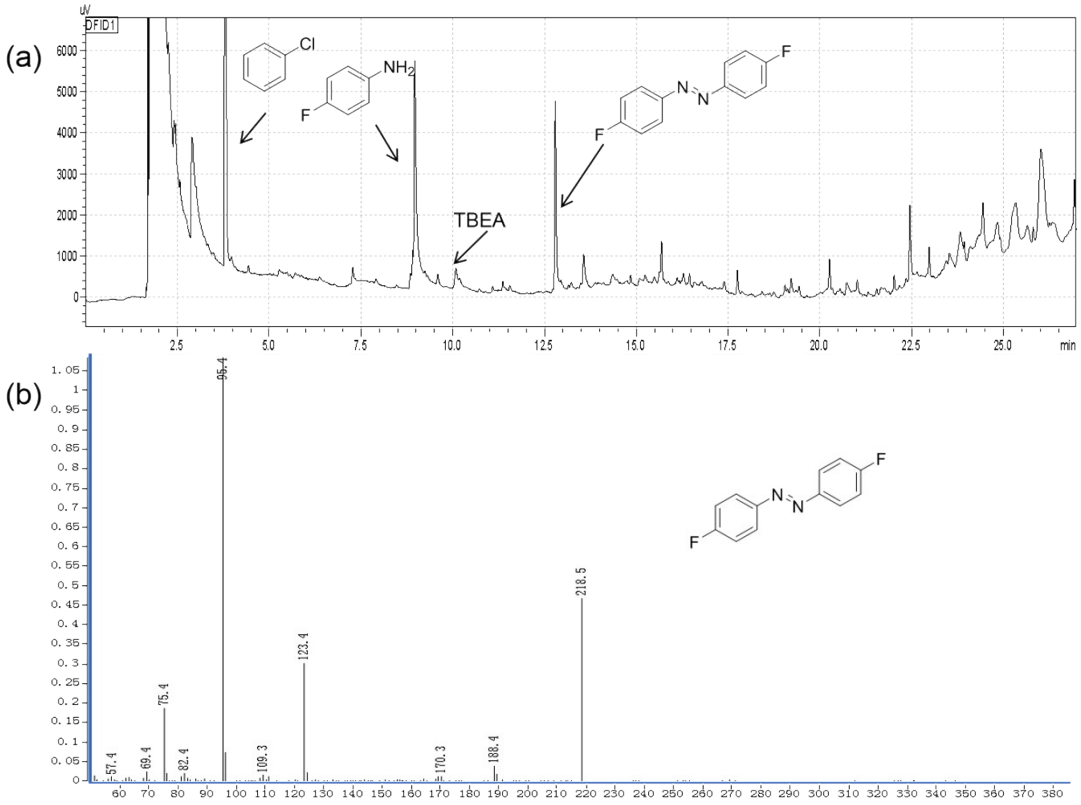


Figure S21. (a) The GC analysis of product (Table 3, entry 4); (b) The MS spectrum of product (Table 3, entry 4).

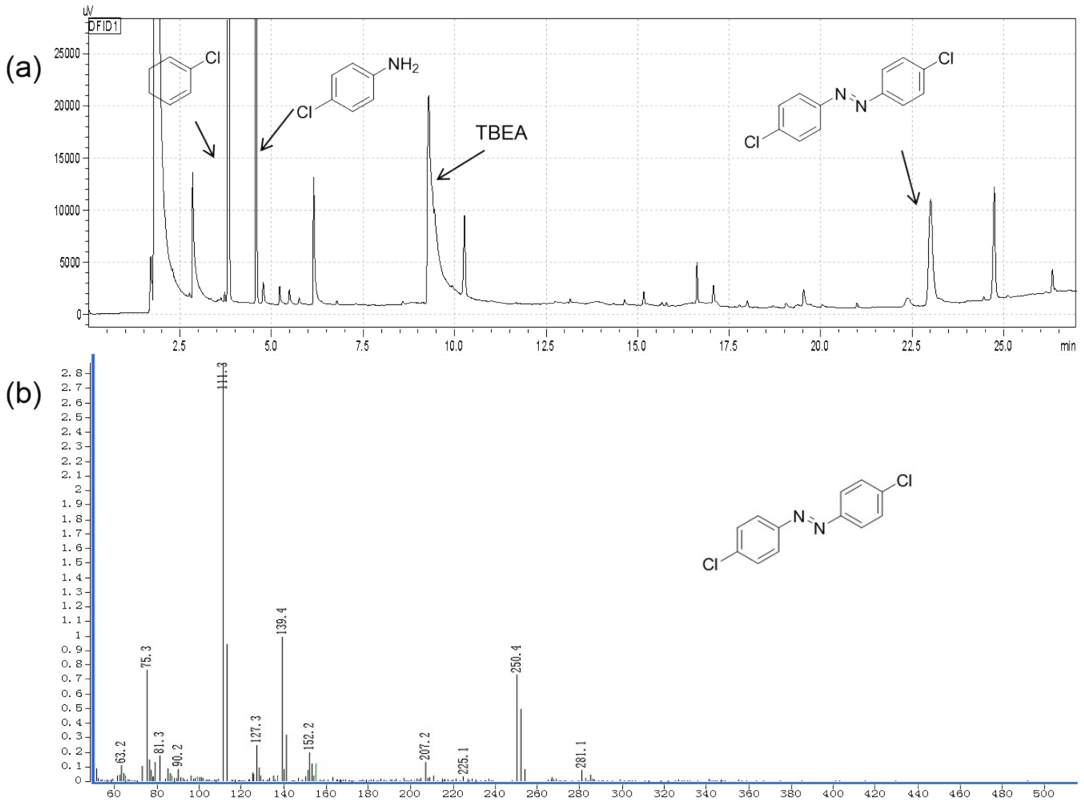


Figure S22. (a) The GC analysis of product (Table 3, entry 5); (b) The MS spectrum of product (Table 3, entry 5).

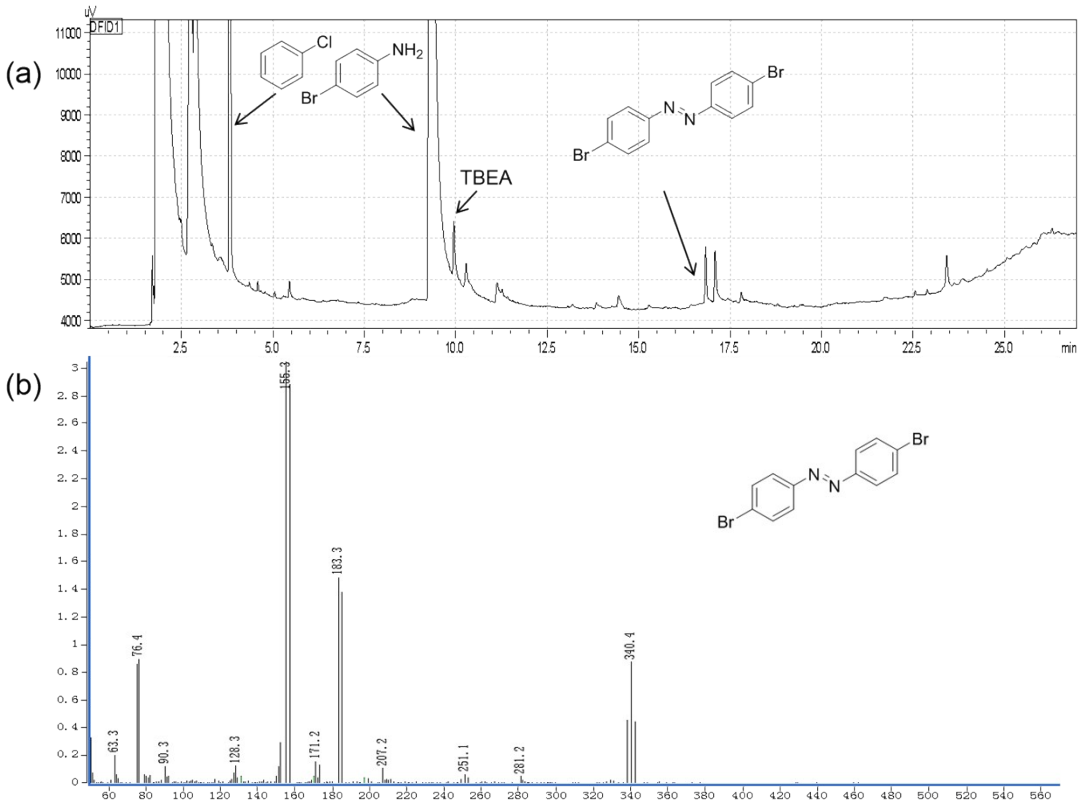


Figure S23. (a) The GC analysis of product (Table 3, entry6); (b) The MS spectrum of product (Table 3, entry 6).

4. Control experiments

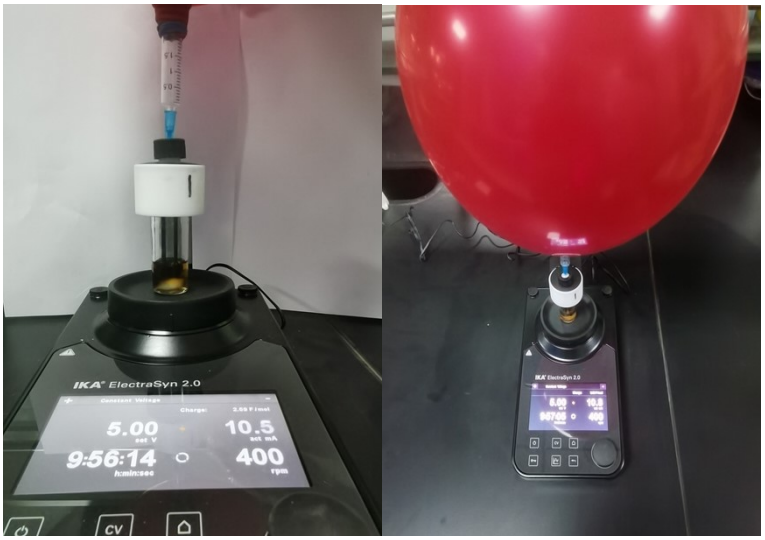


Figure S24. Set-up of control experiments.

Under 5 V constant voltage conditions, a dried ElectraSyn 2.0 vial equipped with a stir bar was loaded with benzylamine (0.25 mmol), TBEA (7 mg,) in CH_3CN (3.0 mL) was stirred at 25 ℃. The tube was equipped with carbon plate (53 mm * 8 mm * 1.5 mm) as the anode and cathode. Remove the air from the reaction vial and fill it with nitrogen to keep it in nitrogen atmosphere. The reaction mixture was stirred and electrolyzed at a constant voltage of 5 V under room temperature for 10 h. After the reaction was completed, the resulting mixture was finally analyzed by GC-MS and GC. The target product 1b was formed in 93% gas chromatography (GC) yield.

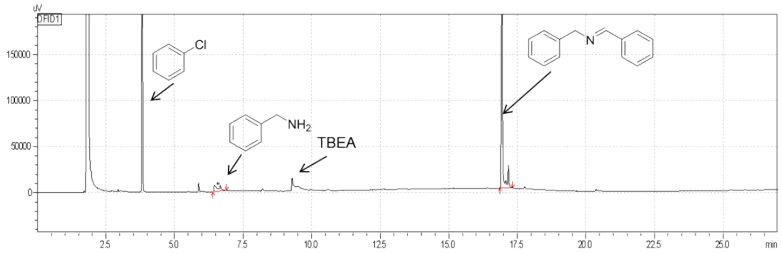


Figure S25. The GC analysis of product (control experiments).