Supporting Information

One-Dimensional Mixed-valence Ce(III)/Ce(IV)-Containing Arsenotungstate for Photocatalytic Synthesis of Quinazolines

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1. General Information

Materials and Methods

The reagents were all obtained from commercial sources and used without further purification.

The FT-IR spectrum was obtained by using a Fourier transform infrared (FT-IR) (4000-500 cm^{-1}) spectrometer (Thermo Nicolet iS5) at 0.5 cm^{-1} resolution and 16 scans. Thermogravimetric analyses (TGA) were performed under N_2 atmosphere on Mettler-Toledo TGA/SDTA 851^e thermal analyzer from 30 to 800 °C. Inductively coupled plasma optical emission spectrum (ICP-OES) data were obtained on an Agilent 725 ICP-OES spectrometer. Powder X-ray diffraction (PXRD) was performed on a Bruker D8 Advance diffractometer with Cu Kα radiation (λ = 1.5406 Å) at room temperature. The solid-state ultraviolet diffuse reflection spectrum was acquired on a UV-8000 ultraviolet and visible spectrophotometer equipped with an integrating sphere (Shanghai Metash Instruments Co., Ltd). The GC analysis was performed on an Agilent 7890B equipped with a capillary column (HP-5, 30 m × 0.25 μm) using a flame ionization detector. The GC-MS was recorded on Agilent 7890B-7000D. Flash column chromatography was performed using silica gel of 200-300 mesh.

X-ray Crystallography

The single crystal X-ray diffraction data were collected on a Bruker D8 Smart Apex II diffractometer with graphite monochromated Mo Kα radiation (λ = 0.71073 Å). Intensities were collected by *ω*-scan and reduced on *APEX 3,* and a multi-scan absorption correction was applied.[1] The structures were solved and refined on *Olex2* using the *SHELX* package.[2] Parameters of the crystal data collection and refinement are given in Table S1. The CCDC number is 2522416.

2. Experimental

Synthesis of $Ce_8As_4W_{36}$

A 1 mol/L aqueous solution of $NaAsO_2$ was prepared using deionized water (Solution A). Na_2WO_4 (1.400 g, 4.24 mmol) and $CH_3NH_2 \cdot HCl$ (0.472 g, 5.79 mmol) were dissolved in 20 mL of deionized water (Solution B). First, 0.5 mL of solution A was added to solution B using a pipette. The mixture was sonicated to facilitate dissolution, yielding a clear solution. The pH was then adjusted to 4.0 with 6 M HCl, followed by stirring for 10 min. $(NH_4)_2Ce(NO_3)_6$ (0.360 mmol, 0.200 g) was subsequently added to the solution, and the pH was adjusted to 2.0 after the solids were completely dissolved. The yellow solution was stirred for another 30 min before being filtered. The clear filtrate was then left to evaporate at room temperature. Yellow crystals could be obtained after about four weeks (30.2% yield based on W). Elemental analysis (%) for $\mathbf{Ce_8As_4W_{36}}$: calculated: Na, 3.66; As, 2.65; Ce, 9.92; W, 58.55; FT-IR (cm^{-1}): 3414 (m),1612 (m), 957 (s), 879 (vs), 781 (vs), 683 (vs).

Typical procedure of the photocatalysis reaction

2-Aminobenzylamine (0.2 mmol, 24.4 mg), benzaldehyde (0.3 mmol, 31.8 mg), $\mathbf{Ce_8As_4W_{36}}$ (0.2 mol%, 4.3 mg), and dimethyl carbonate (1 mL) were sequentially added to a 25 mL Schlenk flask equipped with a PTFE-coated magnetic stir bar. Subsequently, under an oxygen atmosphere, the reaction flask was placed under irradiation from a 10 W, 440 nm LED light source and stirred at room temperature for 10 h. At the end of the reaction, the mixture was purified by column chromatography (petroleum ether/EtOAc) to obtain the desired product. The substrate expansion of benzaldehyde was carried out under optimal reaction conditions and the resulting product was purified by column chromatography (petroleum ether/EtOAc).

3. Characterization

Table S1. Crystallographic data and structure refinement of **$Ce_8As_4W_{36}$** (SQUEEZE).

CCDC number	2522416
Empirical formula	$As_4Ce_8Na_3O_{164}W_{36}$
Formula weight	10732.21
Temperature / K	150.0
Crystal system	monoclinic
Space group	C2/m
a[Å]	44.302(9)
b[Å]	25.367(4)
c[Å]	19.725(3)
α[Å]	90
β[Å]	94.821(6)
γ[Å]	90
V[Å^3]	22090(6)
Z	4
ρ_{calcd} [g/cm^{-3}]	3.227
μ [mm^{-1}]	20.905
F (000)	18420.0
Index ranges	$-51 \leq h \leq 52$ $-30 \leq k \leq 26$ $-20 \leq l \leq 23$
Reflections collected	92947
Independent reflections	19924 [R_{int} = 0.0870]
data/restraints/parameters	19924/54/1054
Goodness-of-fit on F^2	1.046
R_1, wR_2 [I > 2σ(I)]	R_1 = 0.0541, wR_2 = 0.1443
R_1, wR_2 [all data]	R_1 = 0.0724wR_2 = 0.1568

$R_1 = \sum||Fo| - |Fc||/\sum|Fo|$, $wR_2 = [\sum w(Fo^2 - Fc^2)^2/\sum w(Fo^2)^2]^{1/2}$

Table S2. Bond valence sum calculations for Ce, As, W atoms in $\mathbf{Ce_8As_4W_{36}}$.

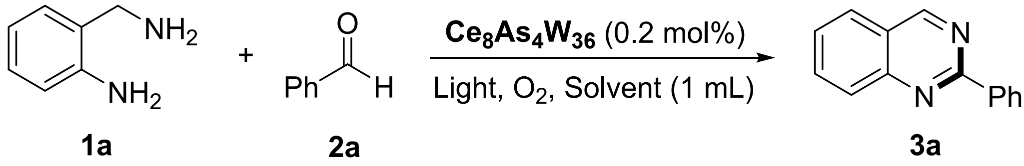
Atom	BVS	Valence	Atom	BVS	Valence
Ce1	3.82	+4	Ce5	3.18	+3
Ce2	3.91	+4	Ce5A	3.18	+3
Ce3	3.98	+4	As1	2.75	+3
Ce4	3.90	+4	As2	2.77	+3
Ce1A	3.82	+4	As3	2.63	+3
Ce2A	3.91	+4	As4	2.75	+3
W1	6.01	+6	W19	6.07	+6
W2	6.05	+6	W20	6.11	+6
W3	5.81	+6	W21	6.11	+6
W4	6.01	+6	W22	5.95	+6
W5	6.02	+6	W23	6.03	+6
W6	5.93	+6	W24	6.04	+6
W7	6.12	+6	W25	5.83	+6
W8	6.19	+6	W26	5.90	+6
W9	6.05	+6	W27	5.97	+6
W10	5.96	+6	W28	5.88	+6
W11	5.98	+6	W29	6.11	+6
W12	6.16	+6	W30	6.06	+6
W13	5.96	+6	W31	5.97	+6
W14	6.22	+6	W32	6.28	+6
W15	6.06	+6	W33	6.03	+6
W16	6.26	+6	W34	5.98	+6
W17	6.02	+6	W35	6.15	+6
W18	6.11	+6	W36	6.25	+6

Bond valence sum (BVS) analysis: The BVS values (V_i) of metal atoms were calculated using the following equation:[3]

$$V_i = \Sigma \exp[(r_0 - r_{ij})/B] \qquad (1)$$

where r_0 is the bond valence parameter for a given atom pair, r_{ij} is the bond length between atoms i and j obtained from the crystal structure.

Table S3. Optimization of the reaction conditions.[a]



Entry	Solvent	Light source (nm)	Time (h)	Yield (%)[b]
1[c]	EA	465	6	16
2	EA	465	6	51
3	DMC	465	6	70
4	PC	465	6	60
5	H_2O	465	6	42
6	EtOH	465	6	35
7	DMC	365	6	51
8	DMC	390	6	57
9	DMC	420	6	68
10	DMC	440	6	76
11	DMC	White LED	6	47
12	DMC	465	8	87
13	DMC	465	10	94
14	DMC	465	12	94
15[d]	DMC	465	10	84
16[e]	DMC	465	10	93

[a] Reaction conditions: **1a** (0.2 mmol), **2a** (0.3 mmol), **$Ce_8As_4W_{36}$** (0.2 mol%) in solvent (1 mL) at room temperature under the irradiation of 10 W LED and O_2 atmosphere for 6 h. [b] The yields were determined by GC with biphenyl as the internal standard. [c] No catalyst. [d] **$Ce_8As_4W_{36}$** (0.1 mol%). [e] **$Ce_8As_4W_{36}$** (0.3 mol%).

Table S4 Comparison of the present catalytic system with other catalysts for this reaction.

Entry	Catalyst (mol%)	Oxidant	Time (h)	Temperature (°C)	Solvent	Yield (%)	TON	Ref
1	**$Ce_8As_4W_{36}$ (0.2)**	**O_2**	**10**	**rt**	DMC	**94**	**470**	**This work**
2	rose bengal (0.5)	O_2	12	rt	DMF	88	176	4
3	Aminoquinolate B,B-diphenyl (1.0)	O_2	10	rt	NMP	80	80	5
4	Fe-Fe_3C@NC-800 (4)	H_2O_2	12	100	THF	96	24	6
5	CuCl (5)	O_2	6	80	MeCN	95	19	7
6	-	IBX	6	rt	MeCN	88	-	8

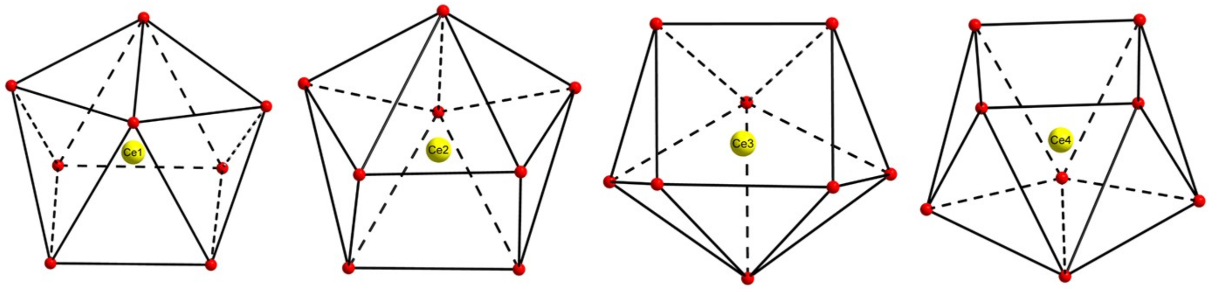


Figure S1 View of the coordination environments of six Ce^{4+} ions.

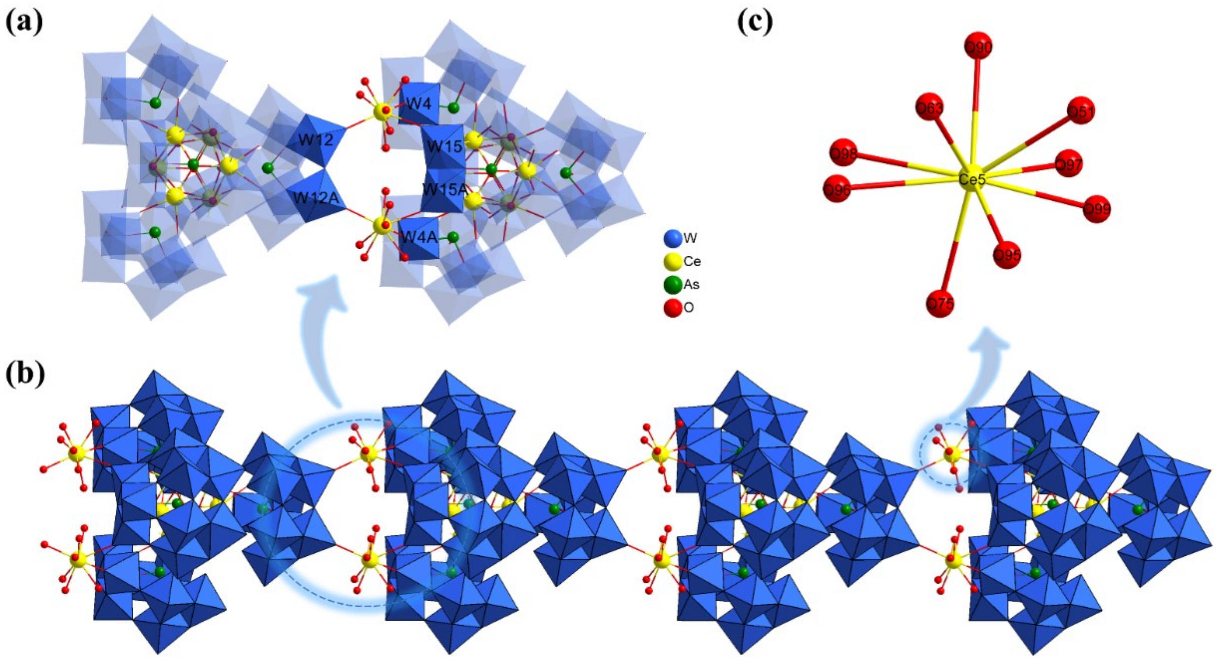


Fig. S2 (a) Two asymmetric units linked by Ce(III) ions; (b) The 1D extended structure of $\mathbf{Ce_8As_4W_{36}}$; (c) Coordination environment of Ce.

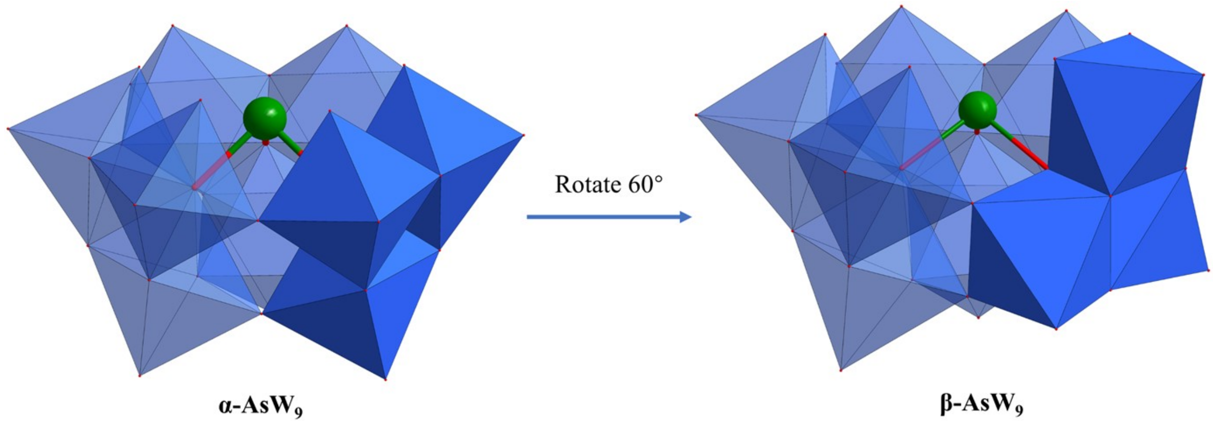


Figure S3 Structural conversion between α- and β-AsW_9 isomers via 60° rotation

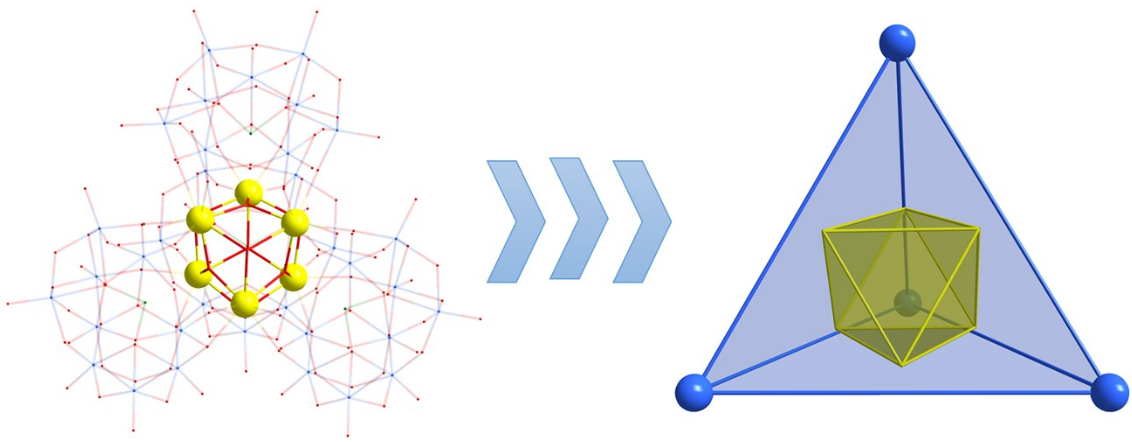


Figure S4 Ball-and-stick model and simplified geometric representation.

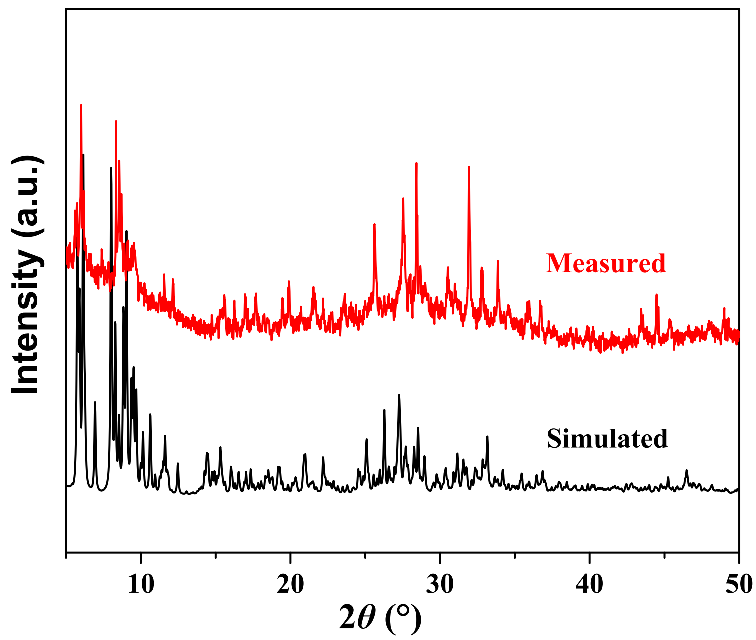


Figure S5 PXRD patterns of $\mathbf{Ce_8As_4W_{36}}$.

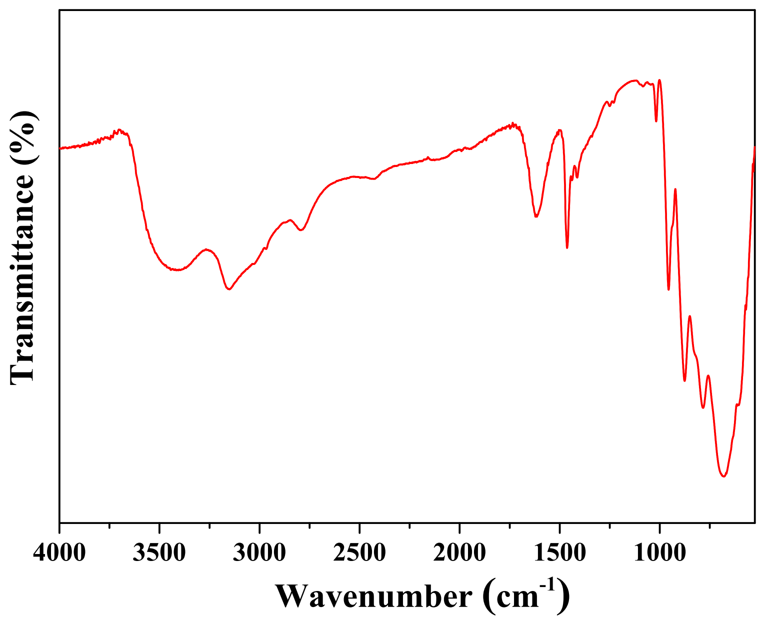


Figure S6 FT-IR spectra of $\mathbf{Ce_8As_4W_{36}}$.

The broad peak around 3414 cm^{-1} is attributed to the ν(O–H) stretching vibration of water molecules and the bending vibration adsorption of water appears at about 1612 cm^{-1}.The peaks that appear in the range of 500 to 1000 cm^{-1} can be attributed to the stretching vibrations of ν(W-O_t), ν(As–O), ν(W–O_b) and ν(W–O_c), and the absorption peaks appearing at 957, 879, 781 and 683cm^{-1}, respectively.

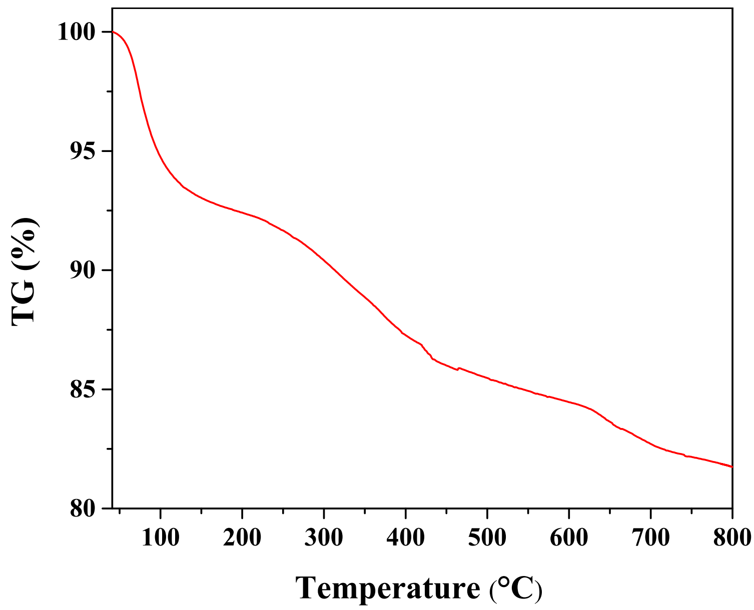


Figure S7 TGA curve of $\mathbf{Ce_8As_4W_{36}}$.

The weight loss at 150 ^{o}C is about 6.98%, which corresponds to the loss of ~45 H_2O molecules. The number of lattice water molecules is about 33.

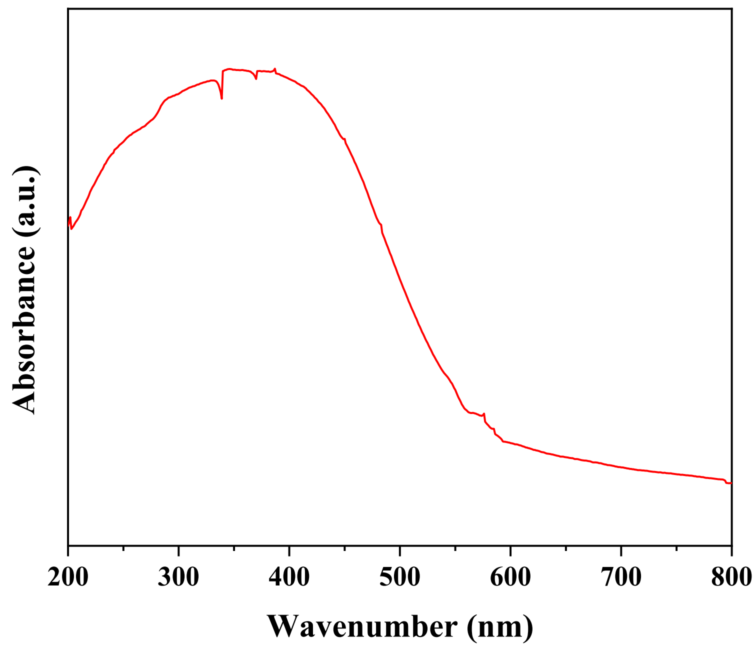


Figure S8 UV-vis spectrum of $\mathbf{Ce_8As_4W_{36}}$

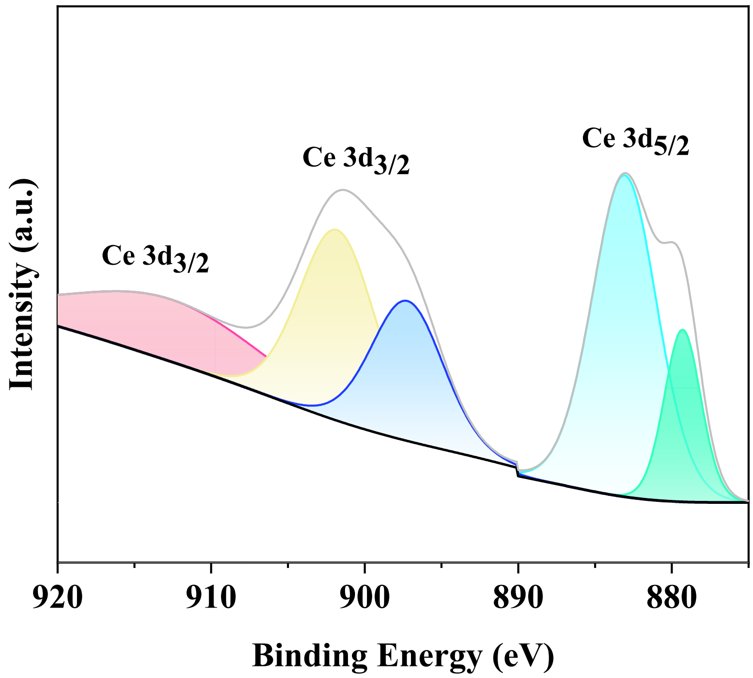


Figure S9 The XPS pattern for Ce $3d_{3/2}$ and Ce $3d_{5/2}$.

XPS studies on $\mathbf{Ce_8As_4W_{36}}$ were conducted to identify the oxidation states of Ce and W atoms. The XPS spectrum for Ce atoms displays three peaks at 897.4, 901.5 and 916.2 eV in the energy region of Ce $3d_{3/2}$, and two peaks at 883.1 and 879.3 eV in the energy region of Ce $3d_{5/2}$. The peaks at 883.1, 897.4 and 901.5 eV are typical for the Ce^{III} ions; whereas the Ce^{IV} ion is labeled with the characteristic peaks at 879.3 and 916.2 eV, illustrating that Ce^{IV} ions in the starting materials are partially reduced to Ce^{III} ions in the synthetic procedure.

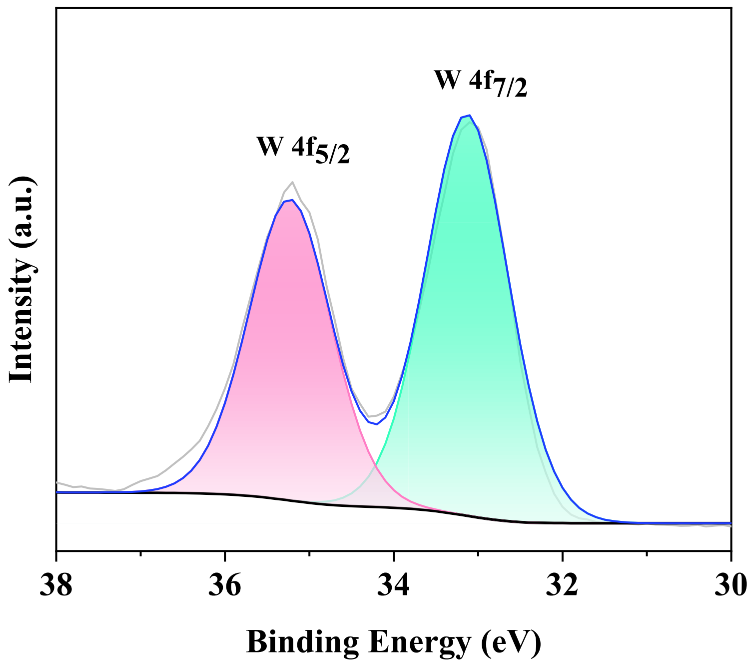


Figure S10 The XPS pattern for W $4f_{5/2}$ and W $4f_{7/2}$.

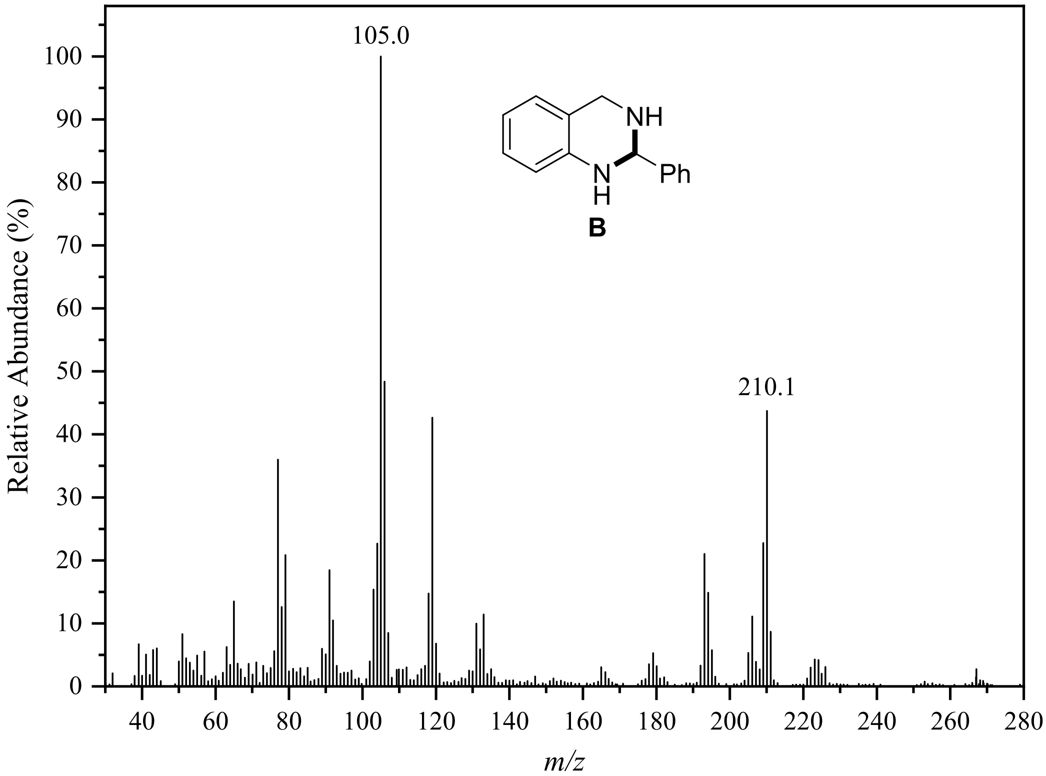


Figure S11 MS spectrum of intermediate **B.**

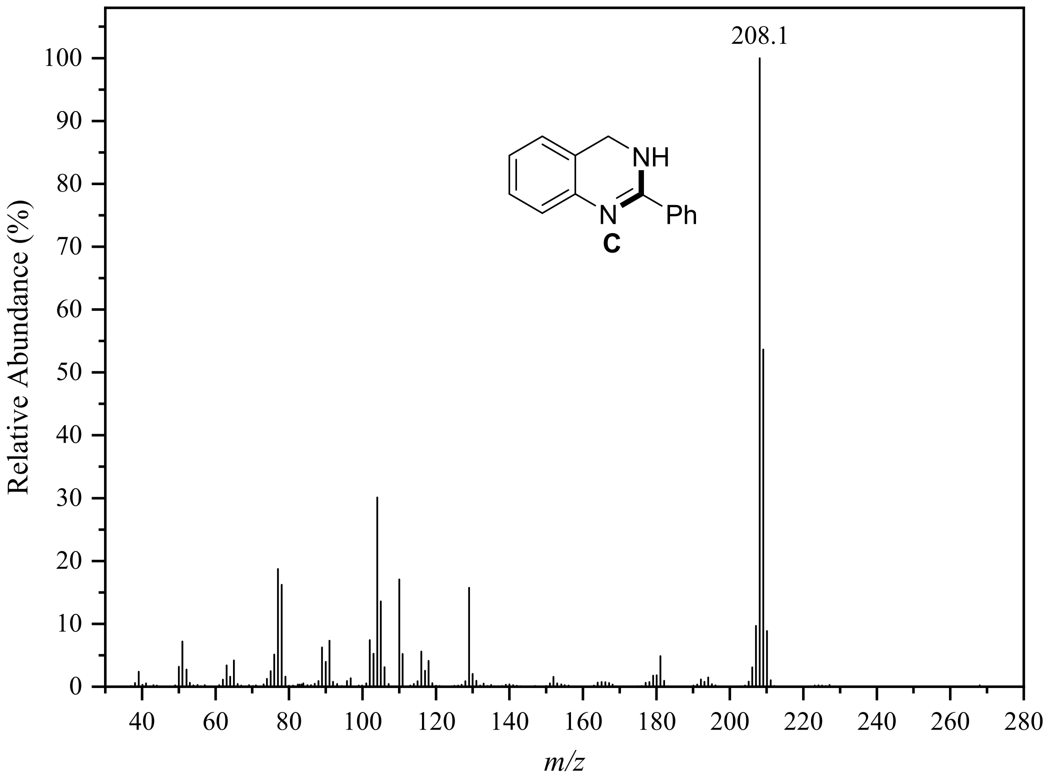


Figure S12 MS spectrum of intermediate **C.**

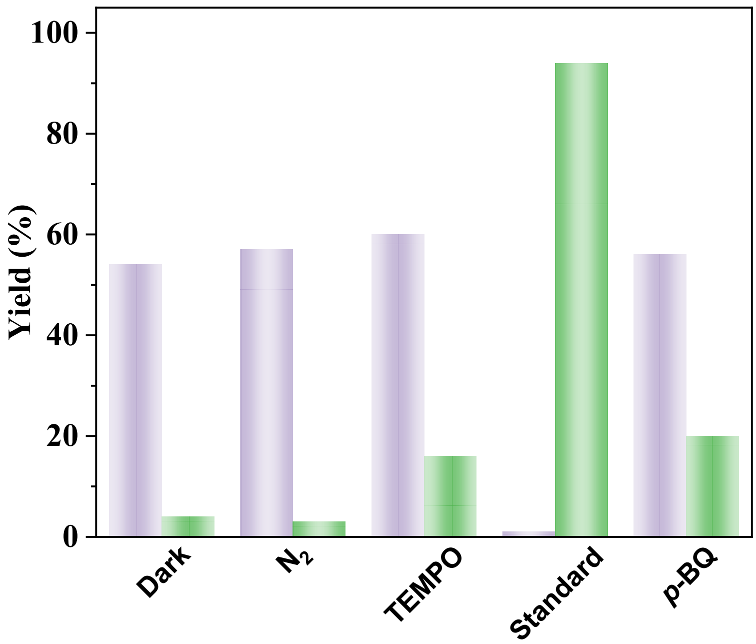
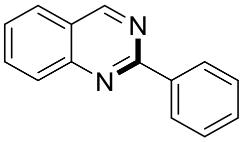


Figure S13 Control experiments.

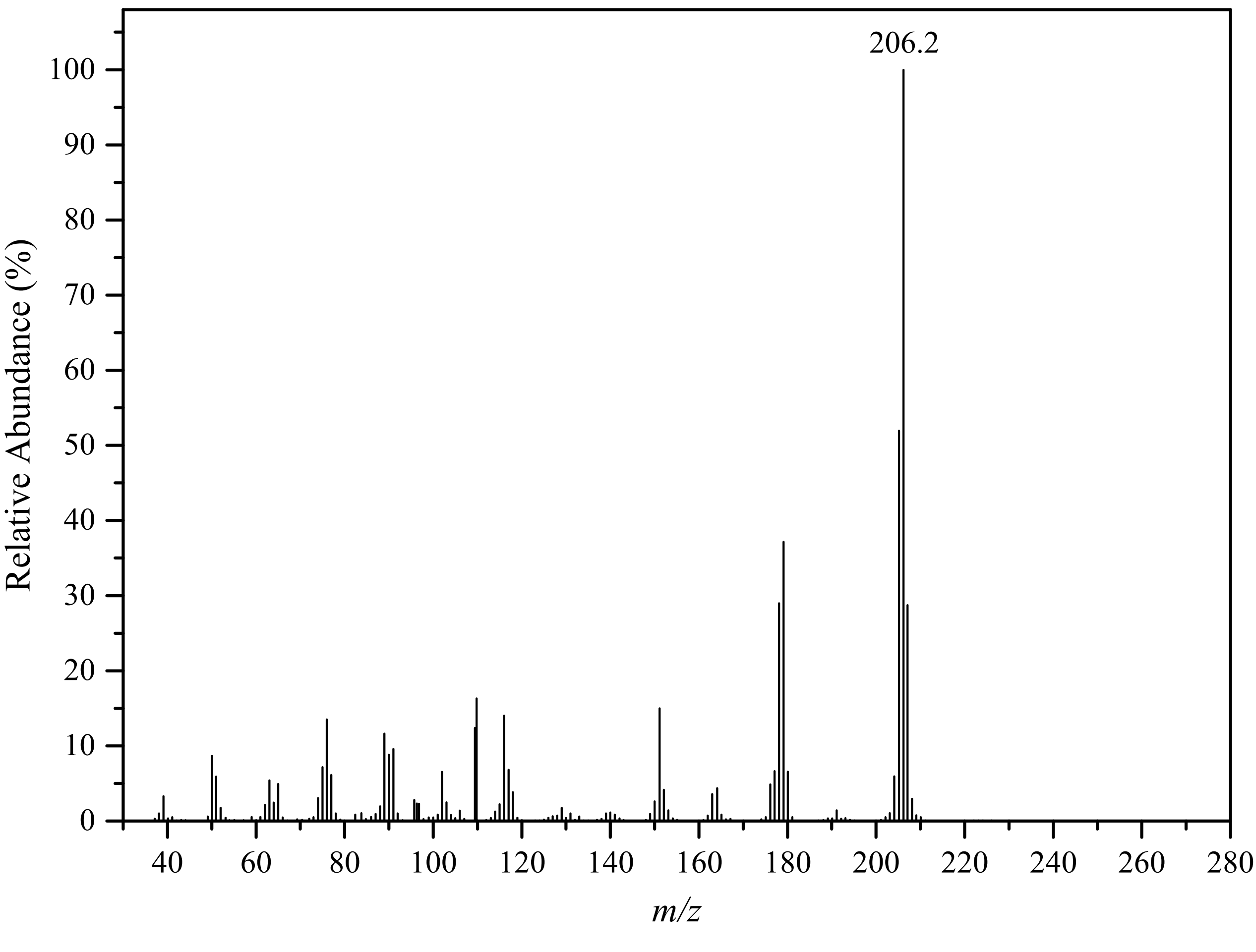
4. Characterization of Products[9]



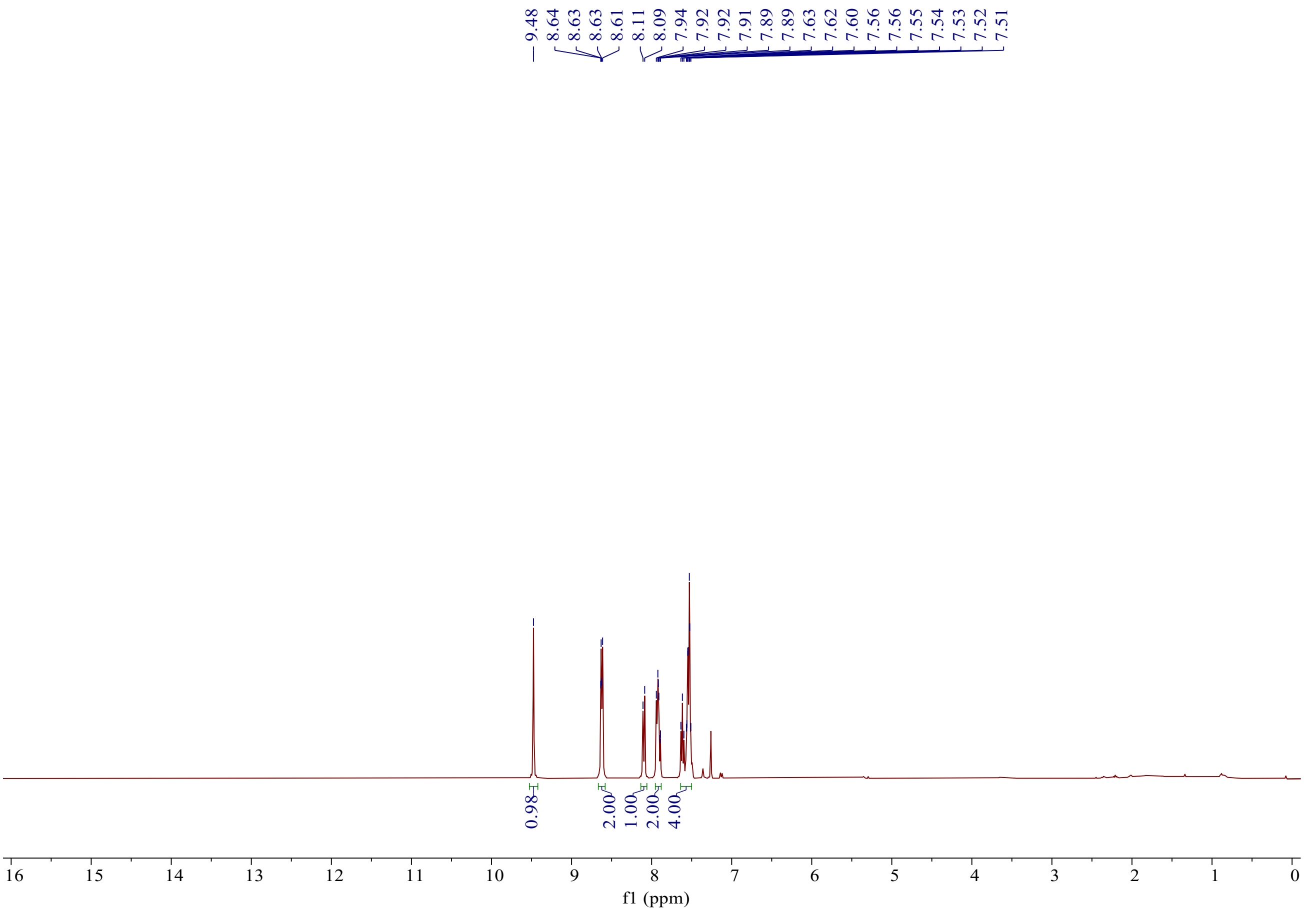
2-phenylquinazoline (3a)

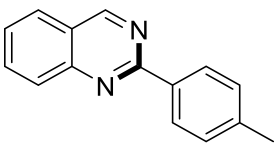
By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), benzaldehyde (0.3 mmol, 31.8 mg), $\mathbf{Ce_8As_4W_{36}}$ (0.2 mol%, 4.3 mg), the product was isolated with 93% yield (38.3 mg).

EI-MS: $C_{14}H_{10}N_2$, m/z (%) = 206.2 (100.0%) [M+].



^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.48 (s, 1H), 8.63 (dd, *J* = 5.7, 3.5 Hz, 2H), 8.10 (d, *J* = 8.5 Hz, 1H), 7.95 – 7.88 (m, 2H), 7.64 – 7.50 (m, 4H).

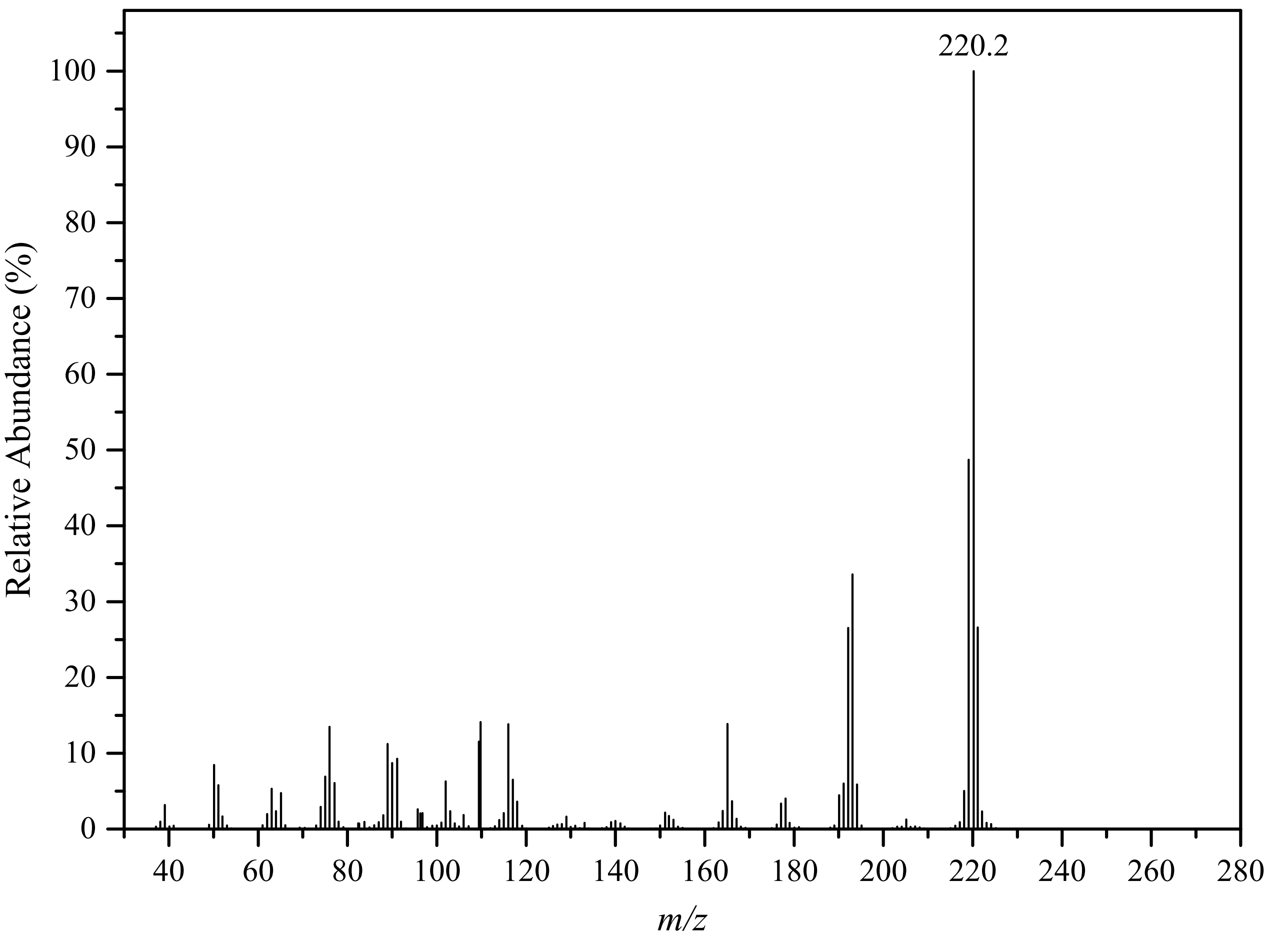


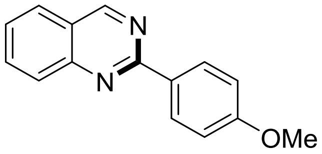


2-(p-tolyl)quinazoline (3b)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-methylbenzaldehyde (0.3 mmol, 36.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 90% yield (39.6 mg).

EI-MS: $C_{15}H_{12}N_2$, m/z (%) = 220.2 (100.0%) [M+].

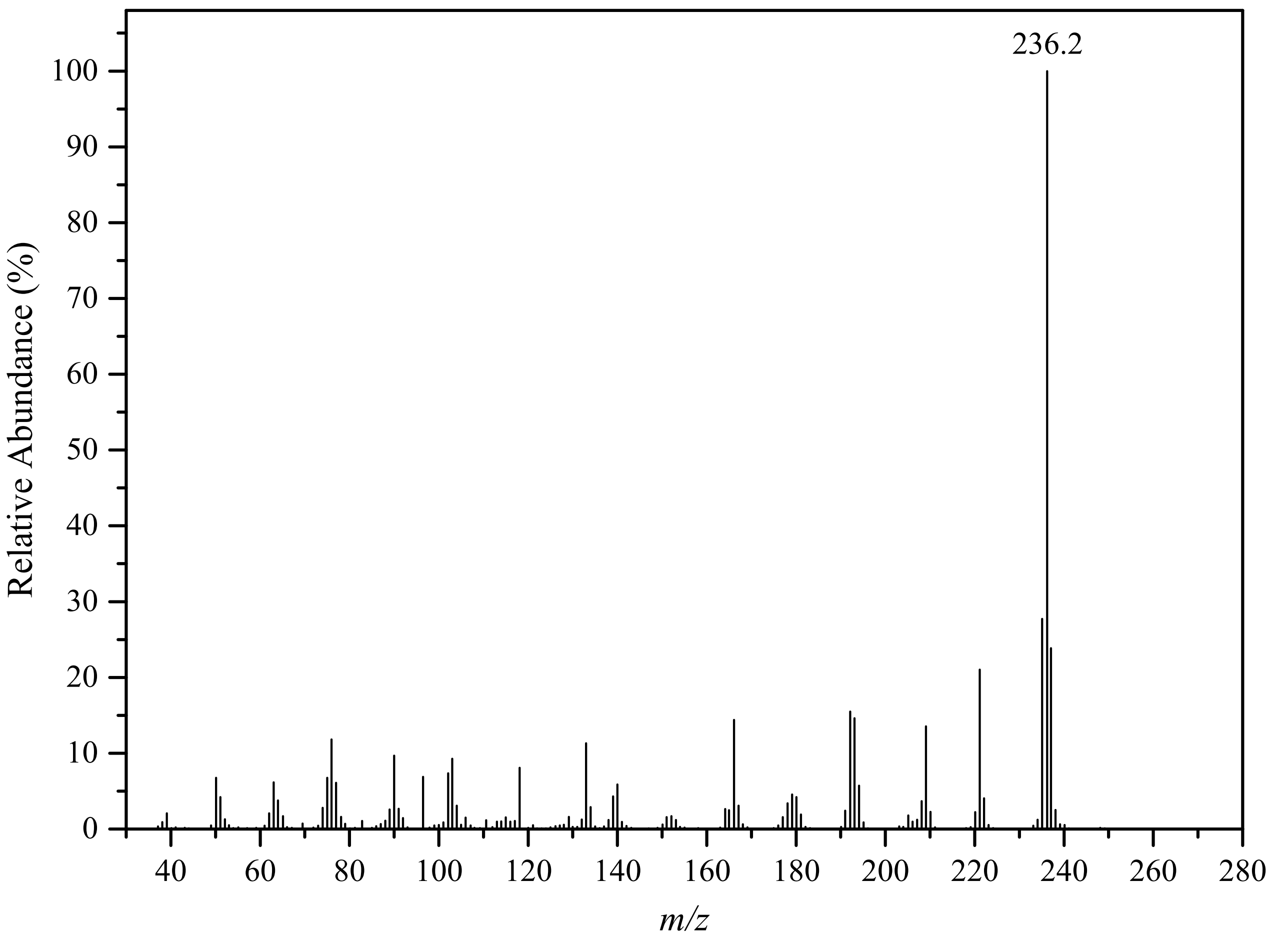


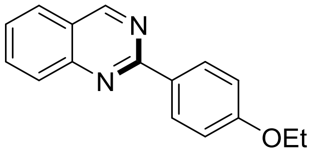


2-(4-methoxyphenyl)quinazoline (3c)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-methoxybenzaldehyde (0.3 mmol, 40.8 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 88% yield (41.5 mg).

EI-MS: $C_{15}H_{12}N_2O$, m/z (%) = 236.2 (100.0%) [M+].

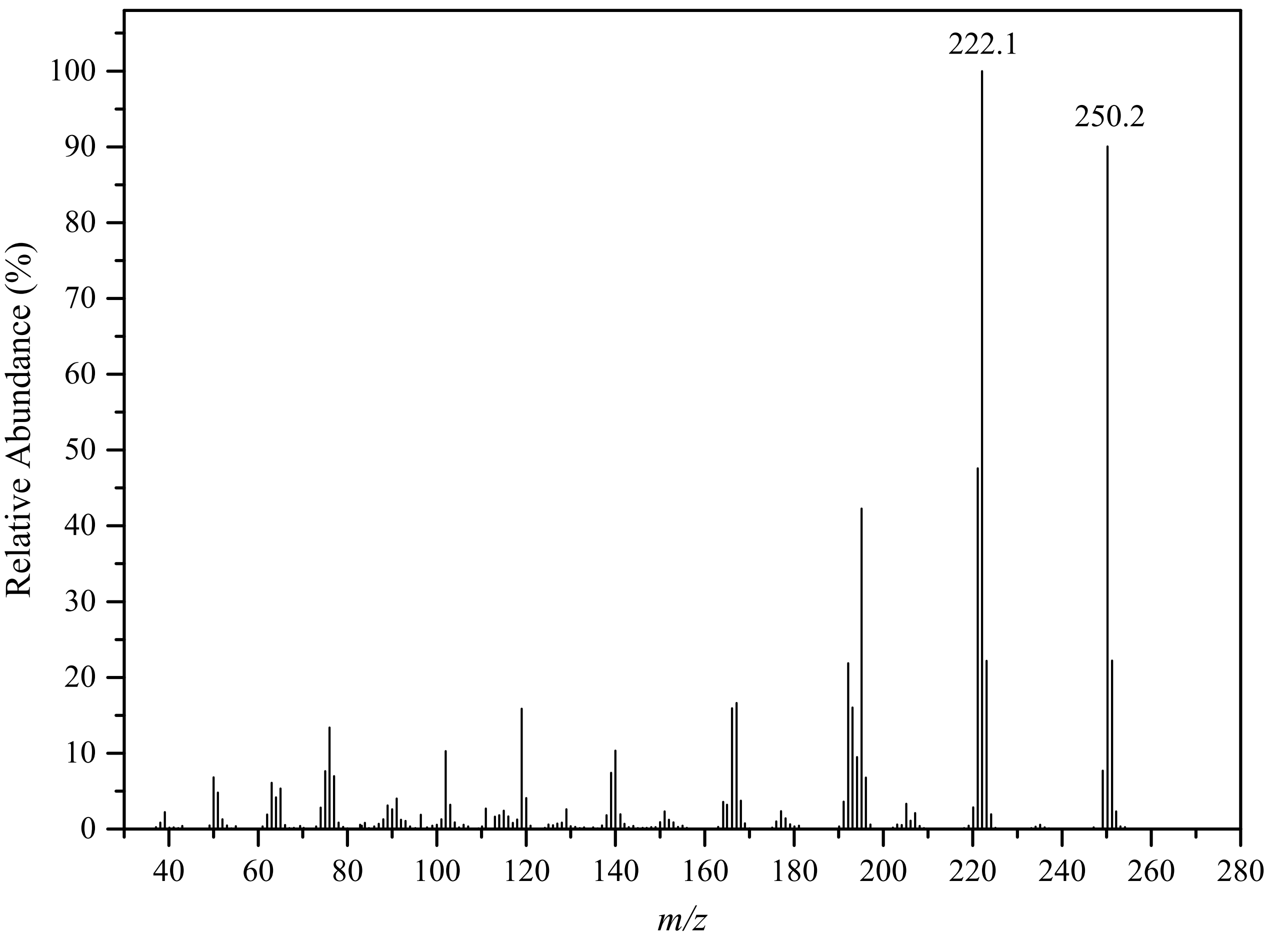


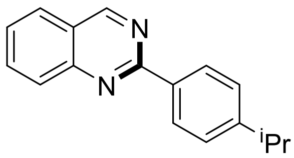


2-(4-ethoxyphenyl)quinazoline (3d)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-ethoxybenzaldehyde (0.3 mmol, 45.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 88% yield (45.5 mg).

EI-MS: $C_{16}H_{14}N_2O$, m/z (%) = 250.2 (90.0%) [M+].

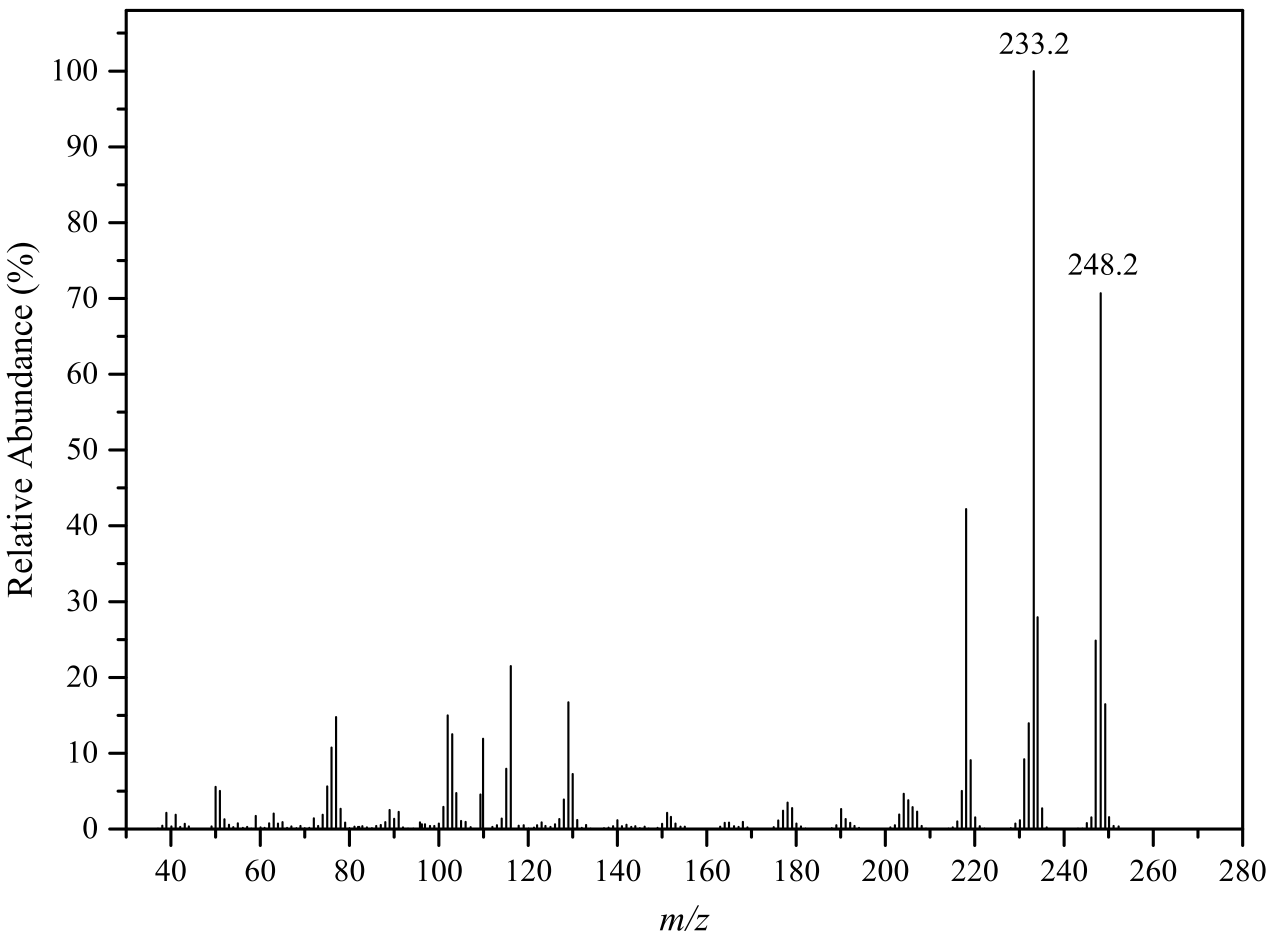


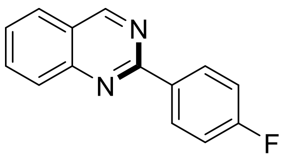


2-(4-isopropylphenyl)quinazoline (3e)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-isopropylbenzaldehyde (0.3 mmol, 44.4 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 91% yield (43.6 mg).

EI-MS: $C_{17}H_{16}N_2$, m/z (%) = 248.2 (70.7%) [M+].

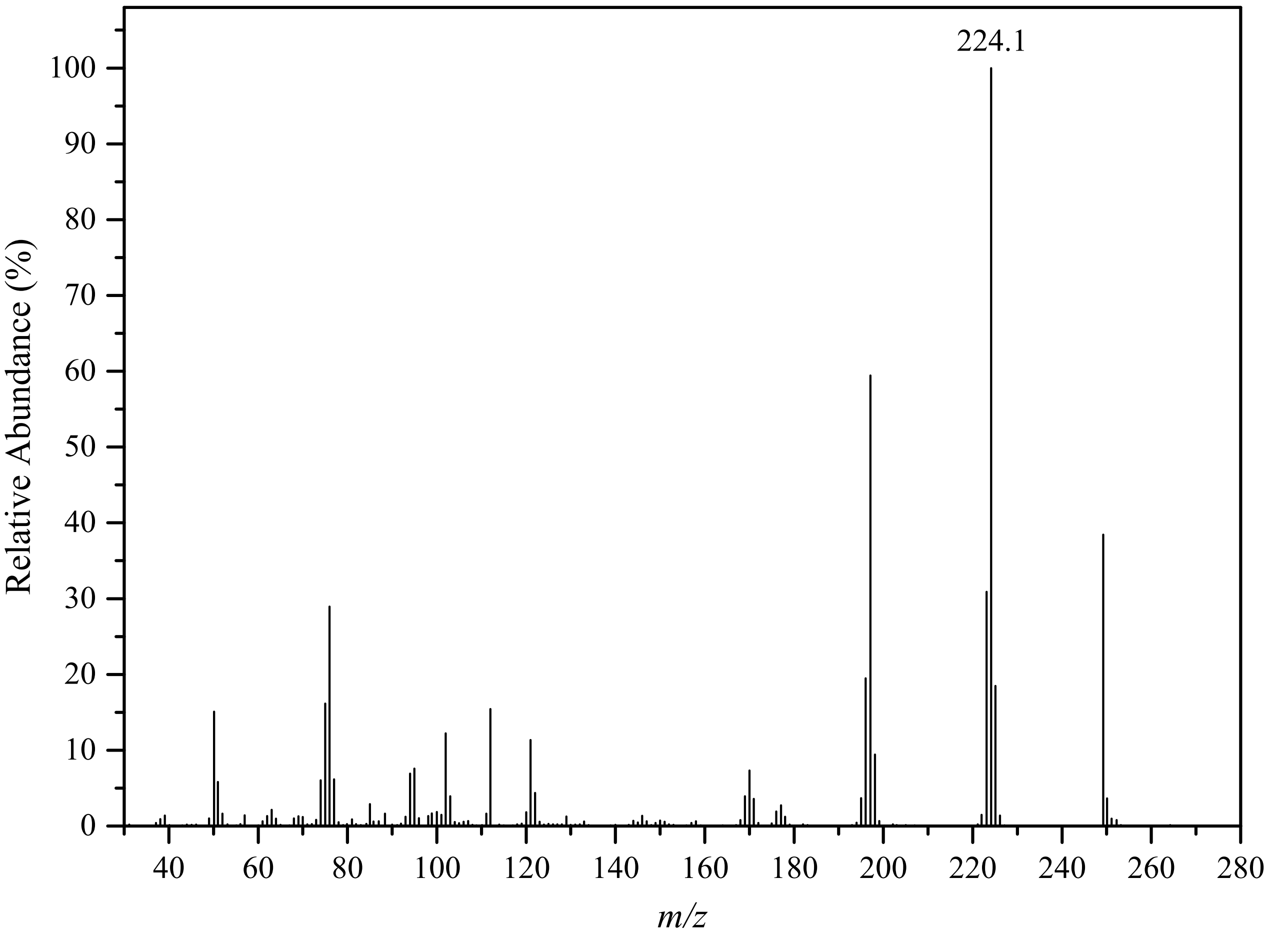


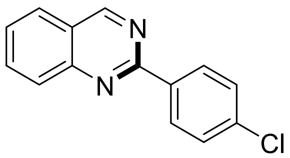


2-(4-fluorophenyl)quinazoline (3f)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-fluorobenzaldehyde (0.3 mmol, 37.2 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 92% yield (41.2 mg).

EI-MS: $C_{14}H_9FN_2$, m/z (%) = 224.1 (100.0%) [M+].

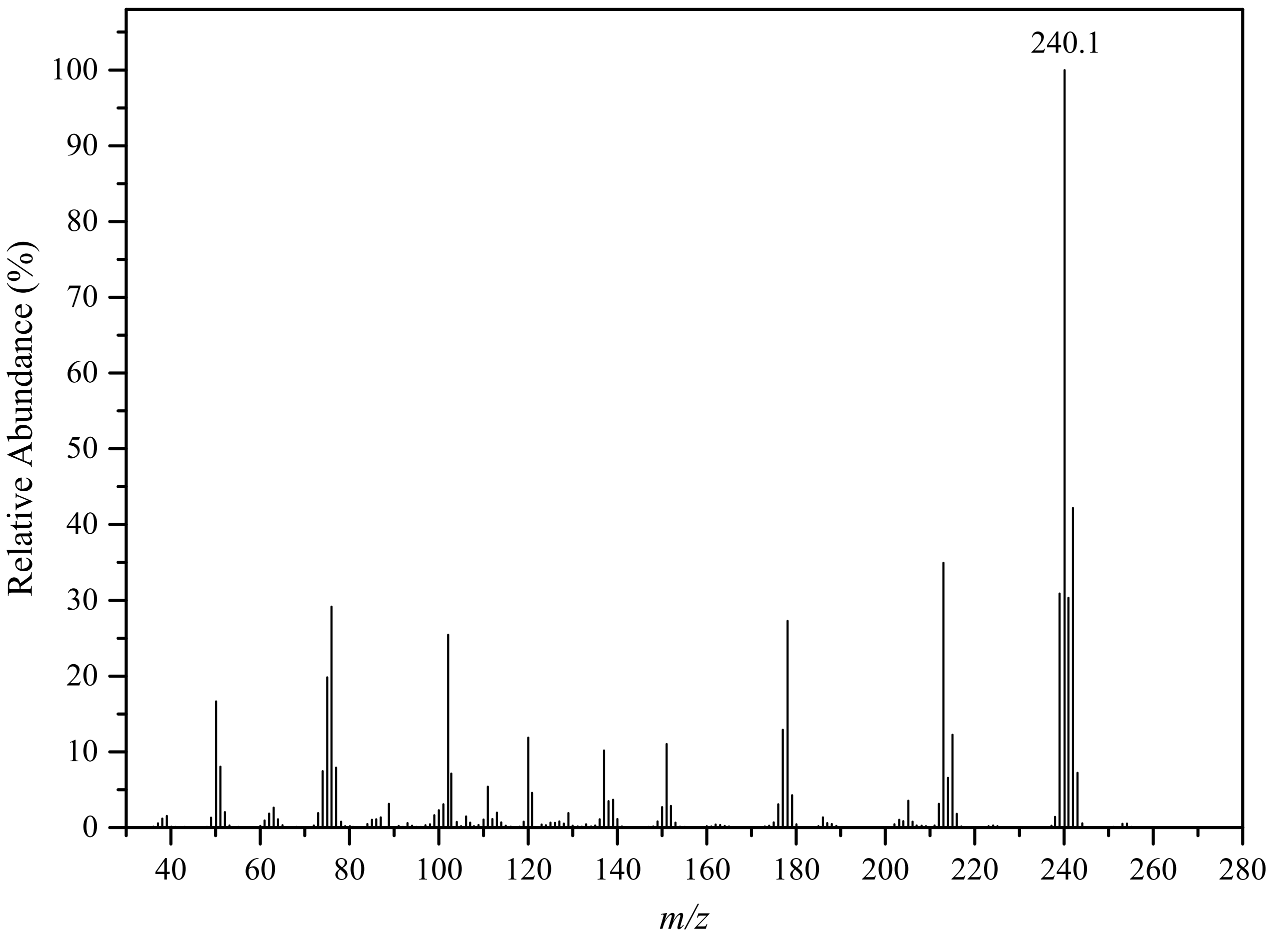


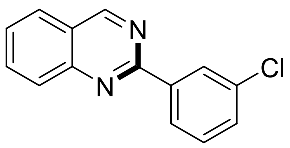


2-(4-chlorophenyl)quinazoline (3g)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-chlorobenzaldehyde (0.3 mmol, 42.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 93% yield (44.6 mg).

EI-MS: $C_{14}H_9ClN_2$, m/z (%) = 240.1 (100.0%) [M+].

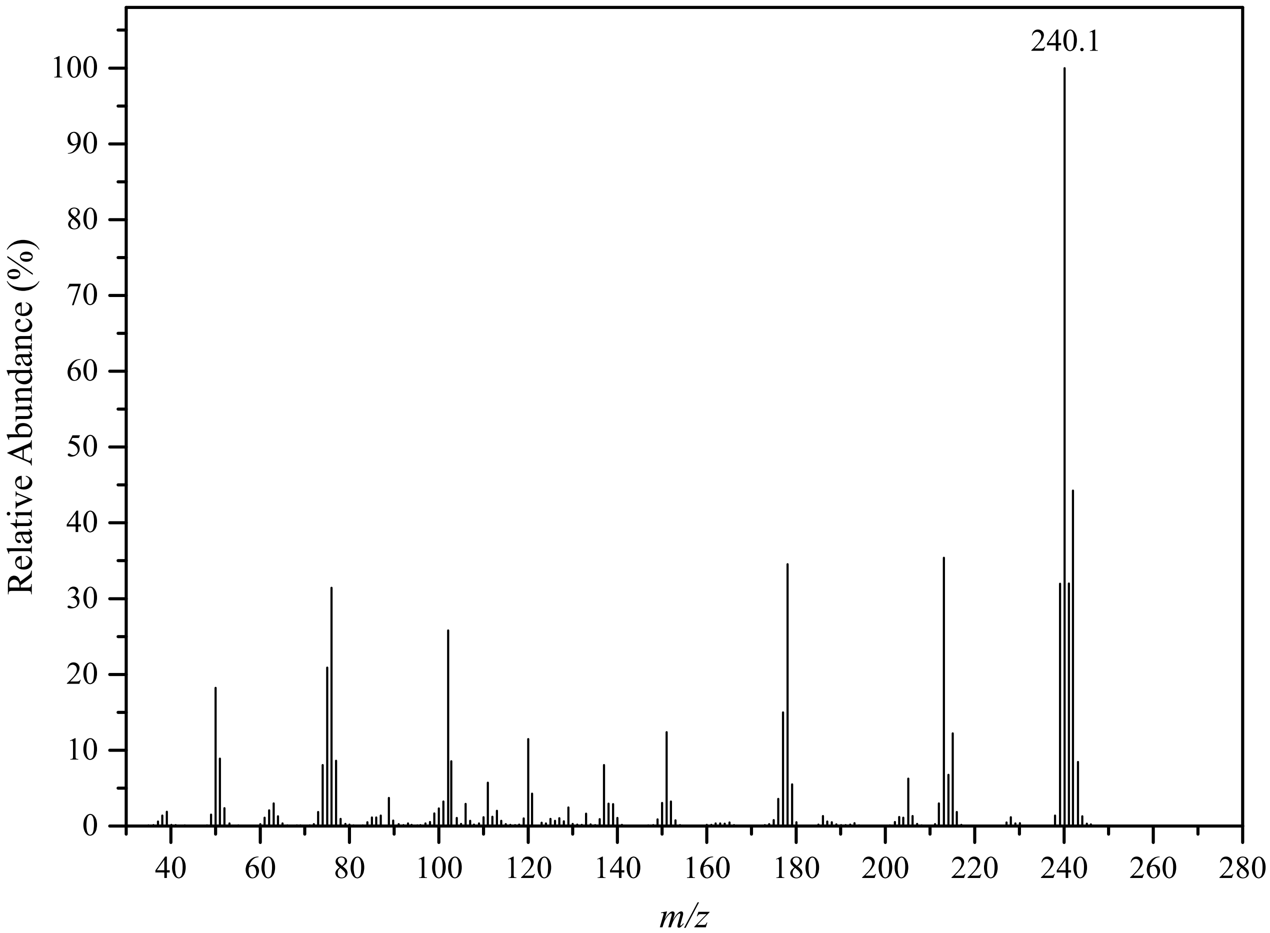


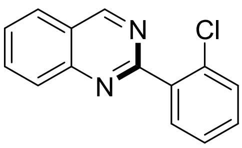


2-(3-chlorophenyl)quinazoline (3h)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 3-chlorobenzaldehyde (0.3 mmol, 42.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 90% yield (43.2 mg).

EI-MS: $C_{14}H_9ClN_2$, m/z (%) = 240.1 (100.0%) [M+].

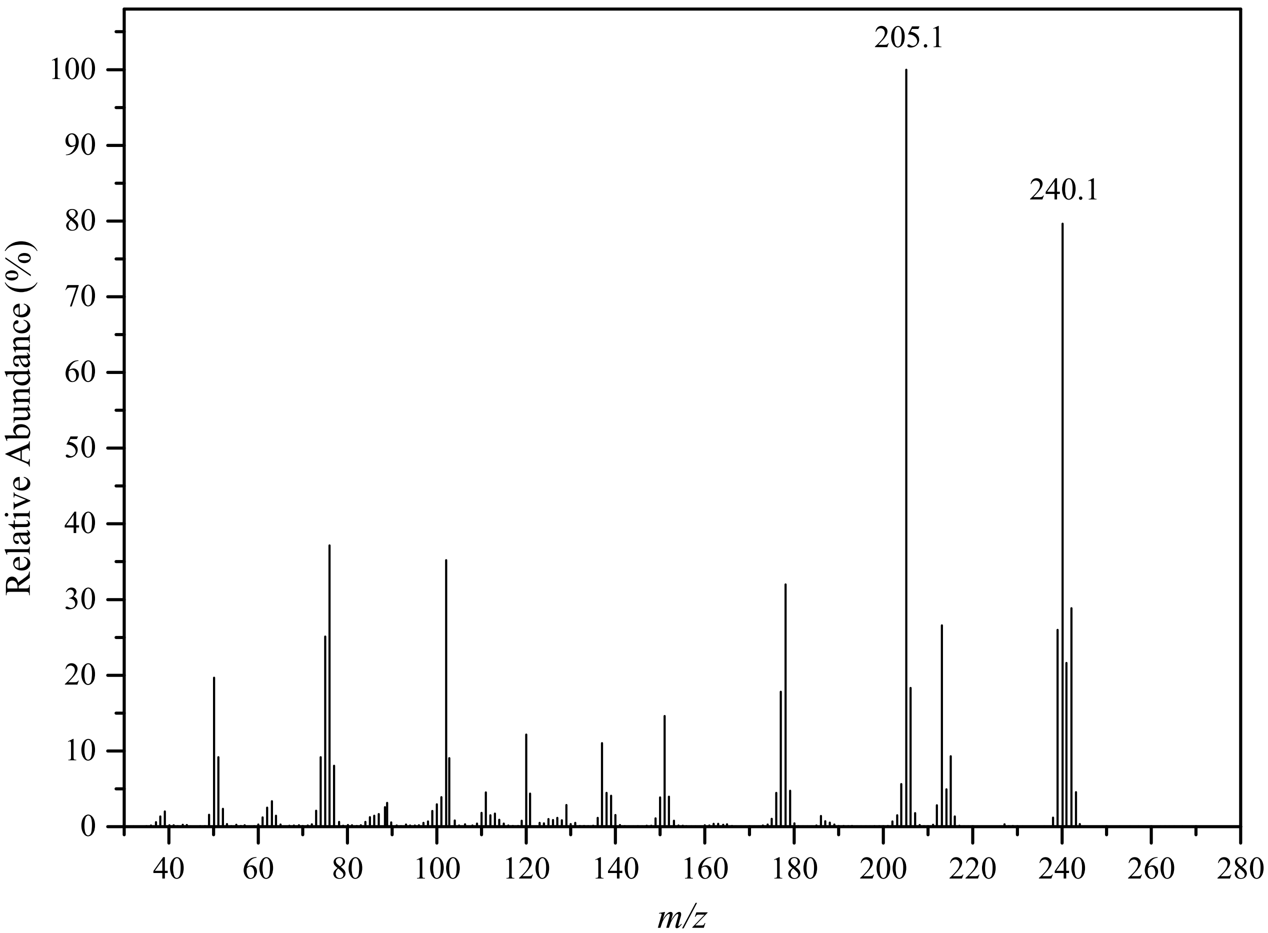


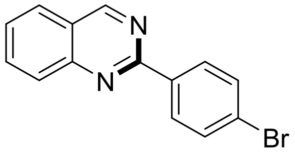


2-(2-chlorophenyl)quinazoline (3i)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 2-chlorobenzaldehyde (0.3 mmol, 42.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 81% yield (38.9 mg).

EI-MS: $C_{14}H_9ClN_2$, m/z (%) = 240.1 (79.6%) [M+].

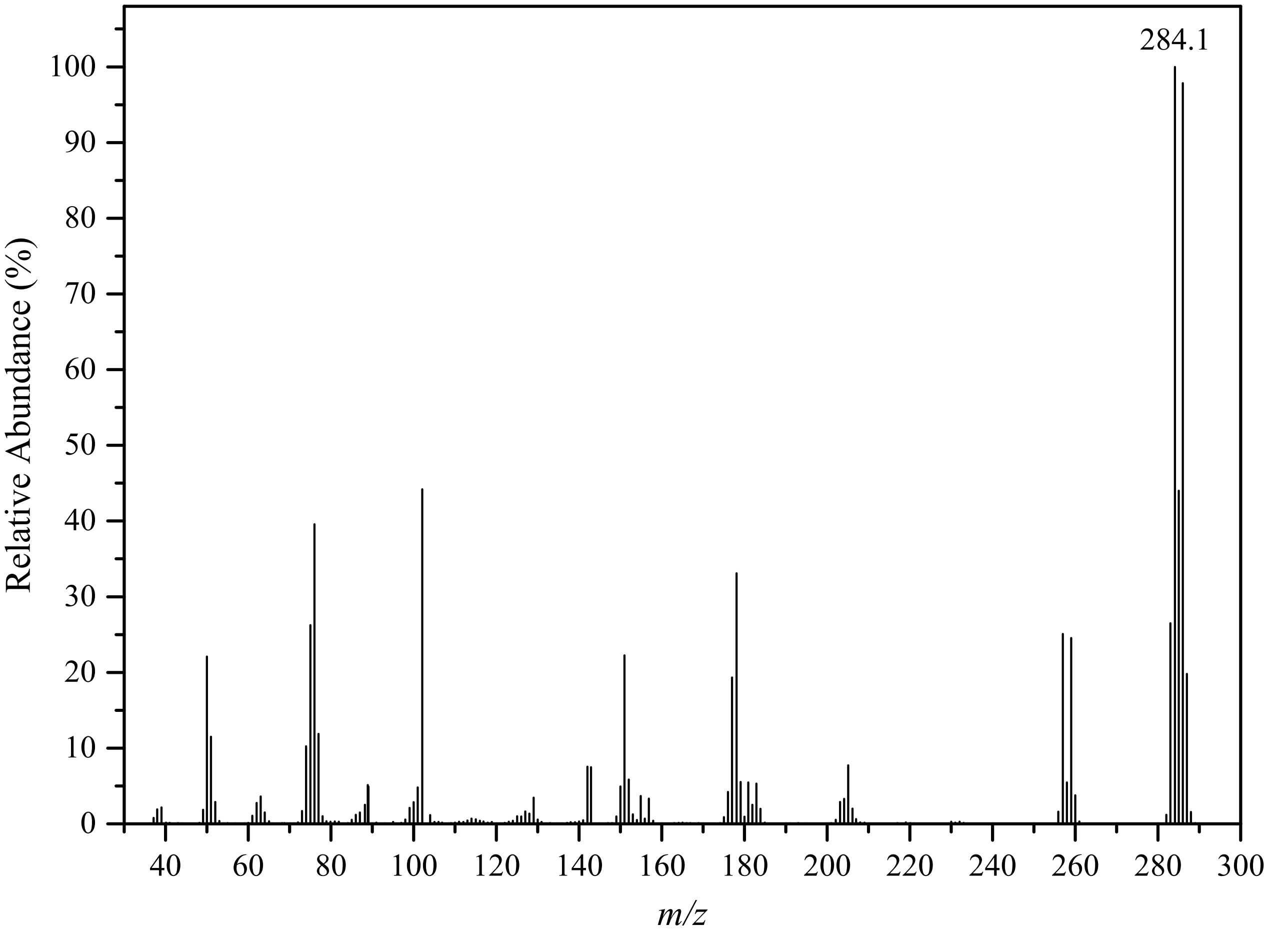


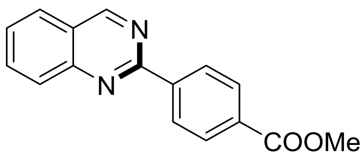


2-(4-bromophenyl)quinazoline (3j)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 4-bromobenzaldehyde (0.3 mmol, 55.5 mg), $\mathbf{Ce_8As_4W_{36}}$ (0.2 mol%, 4.3 mg), the product was isolated with 89% yield (50.6 mg).

EI-MS: $C_{14}H_9BrN_2$, m/z (%) =284.1 (100.0%) [M+].

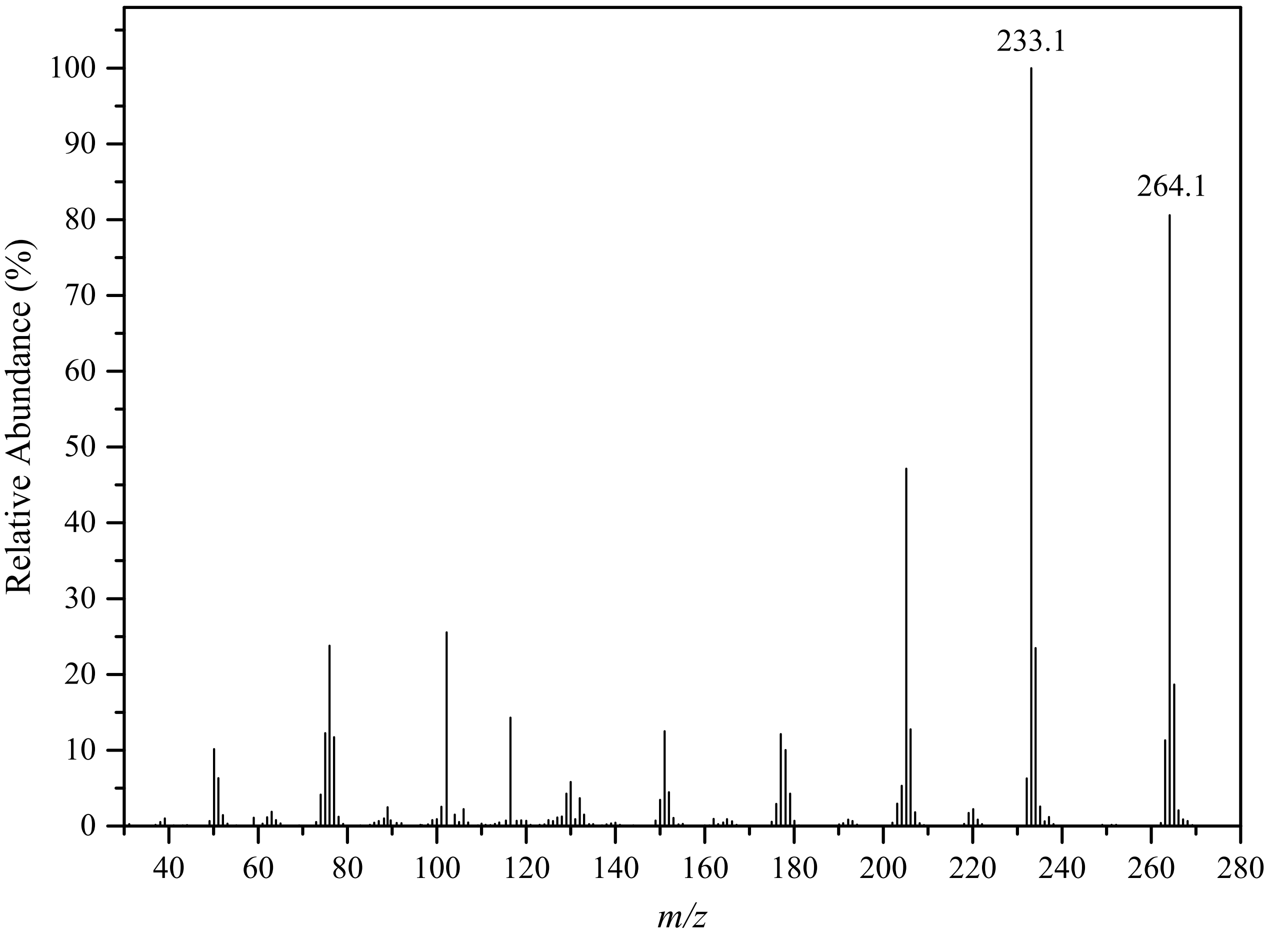


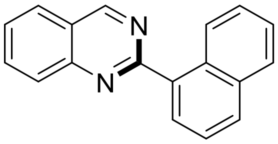


methyl 4-(quinazolin-2-yl)benzoate (3k)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), methyl 4-formylbenzoate (0.3 mmol, 49.2 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 94% yield (49.6 mg).

EI-MS: $C_{16}H_{12}N_2O_2$, m/z (%) = 264.1 (80.6%) [M+].

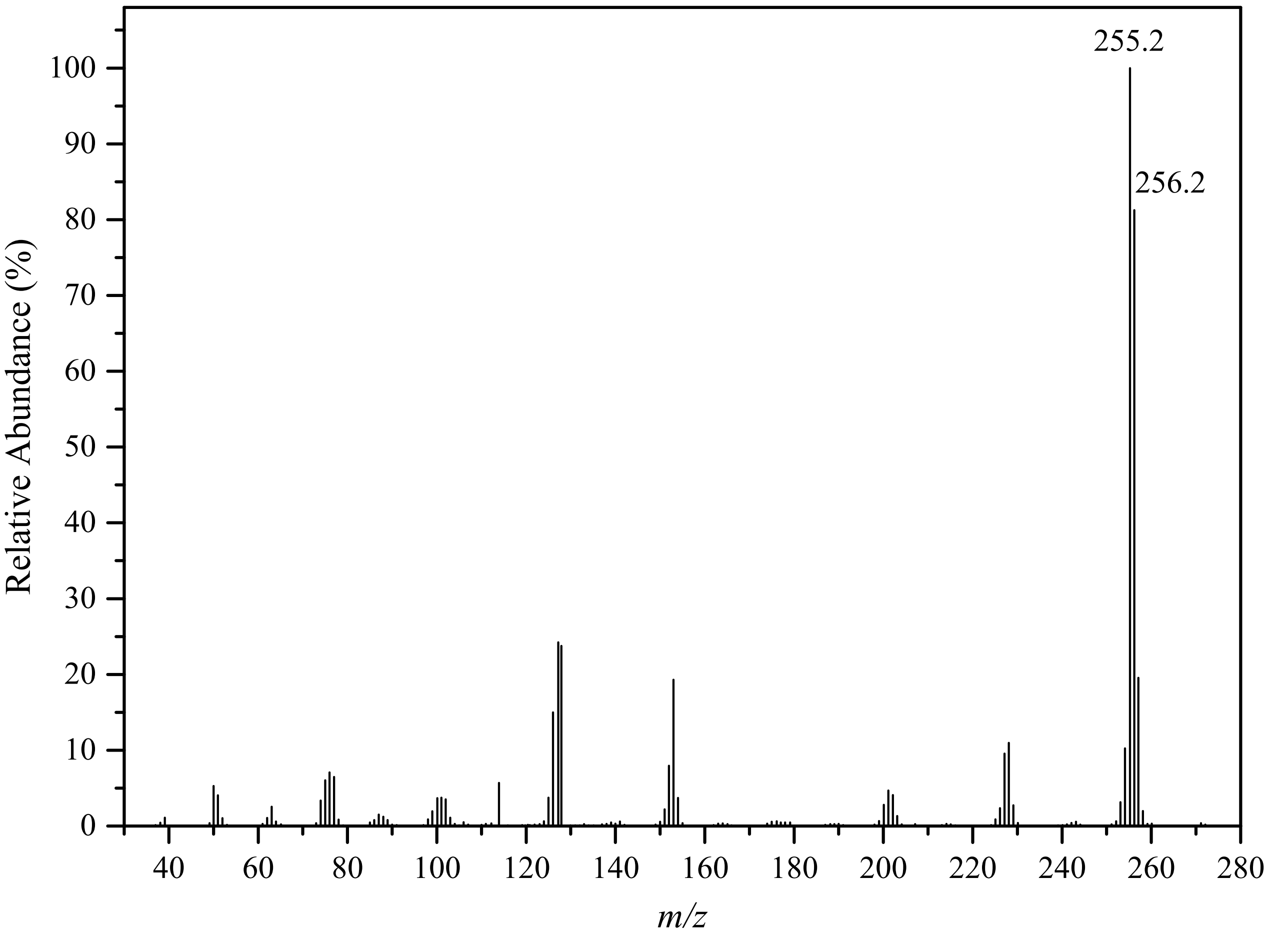


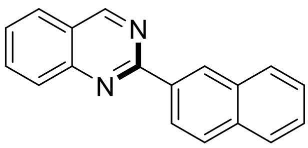


2-(naphthalen-1-yl)quinazoline (3l)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 1-naphthaldehyde (0.3 mmol, 46.8 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 92% yield (47.1 mg).

EI-MS: $C_{18}H_{12}N_2$, m/z (%) = 256.2 (81.2%) [M+].

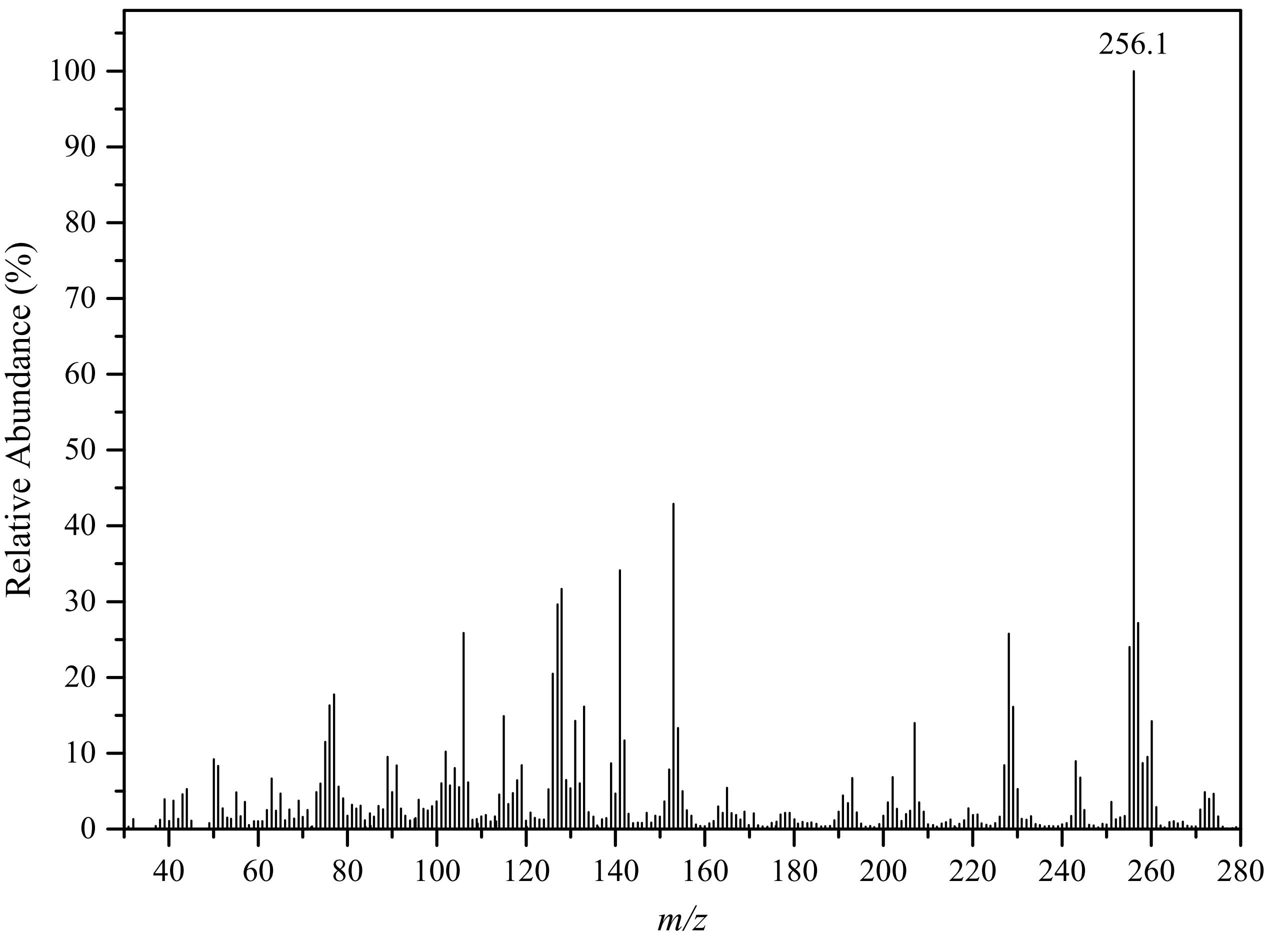


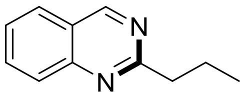


2-(naphthalen-2-yl)quinazoline (3m)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), 2-naphthaldehyde (0.3 mmol, 46.8 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 90% yield (46.1 mg).

EI-MS: $C_{18}H_{12}N_2$, m/z (%) = 256.1 (100.0%) [M+].

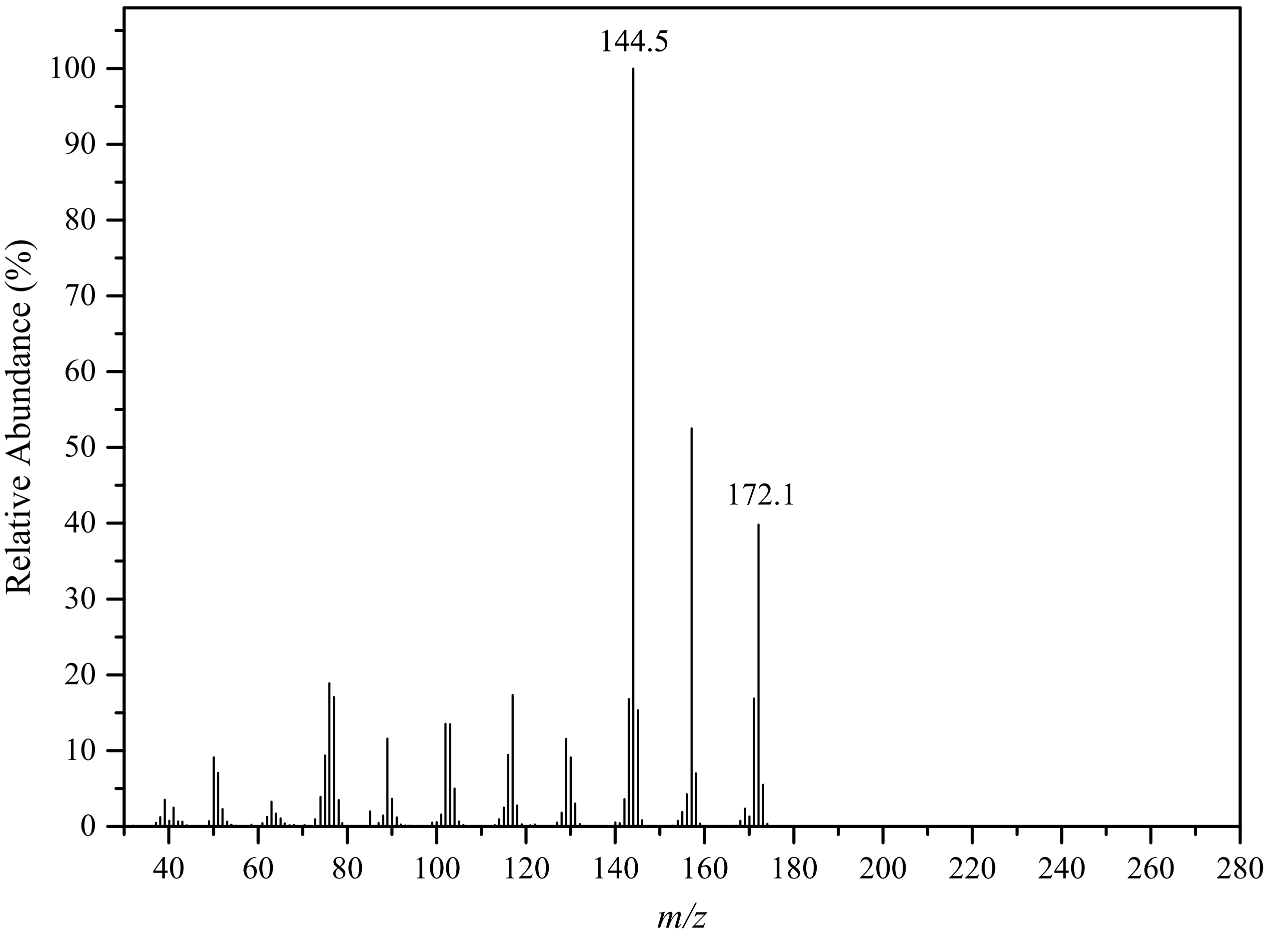


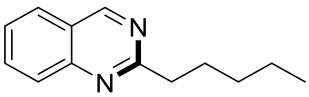


2-propylquinazoline (3n)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), butyraldehyde (0.3 mmol, 21.6 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 86% yield (29.6 mg).

EI-MS: $C_{11}H_{12}N_2$, m/z (%) = 172.1 (39.8%) [M+].

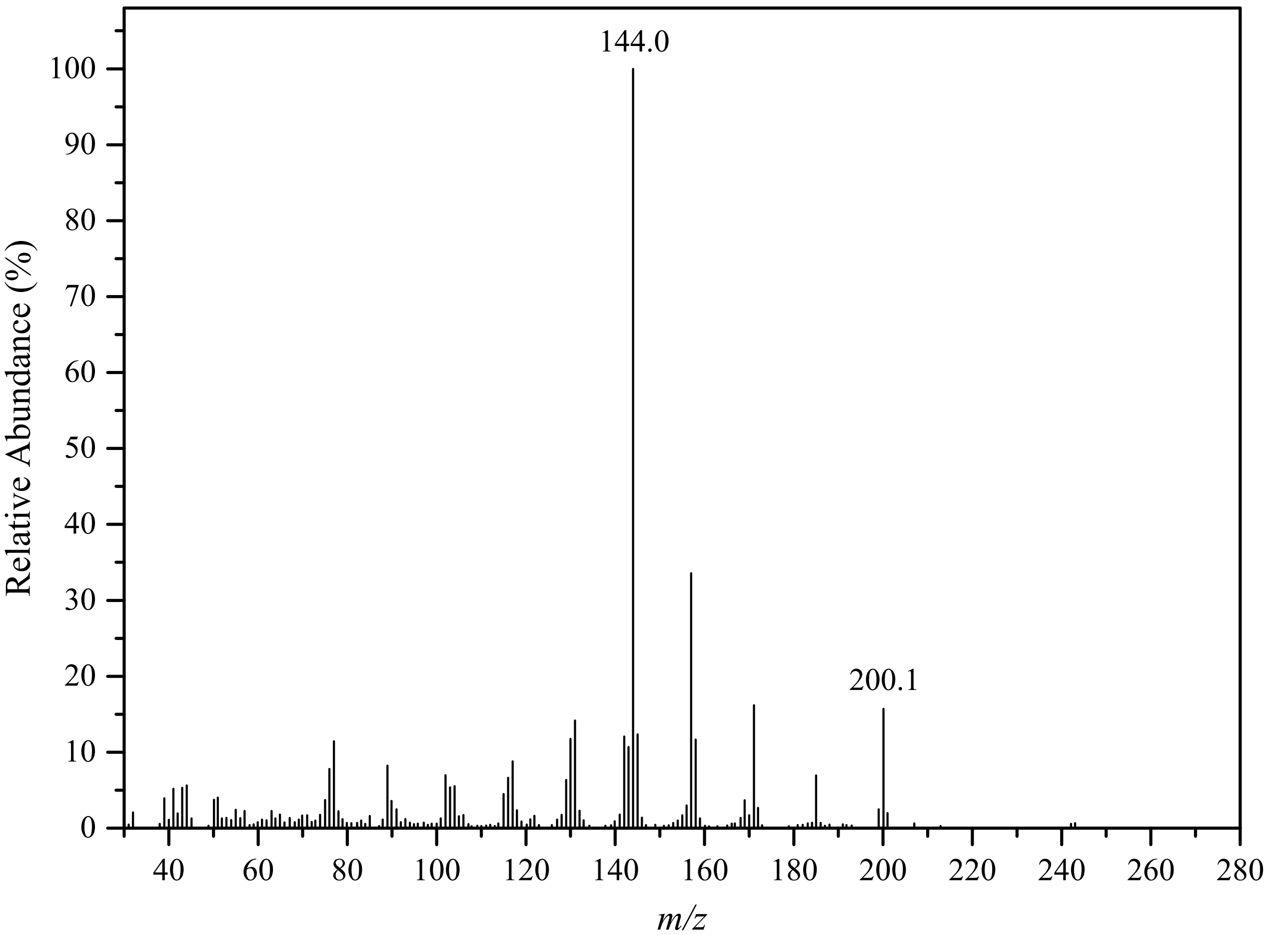


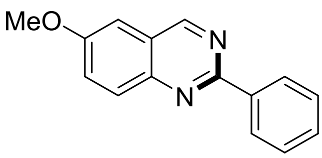


2-pentylquinazoline (3o)

By following the typical procedure with 2-aminobenzylamine (0.2 mmol, 24.4 mg), hexanal (0.3 mmol, 30.0 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 84% yield (33.6 mg).

EI-MS: $C_{13}H_{16}N_2$, m/z (%) = 200.1 (15.7%) [M+].

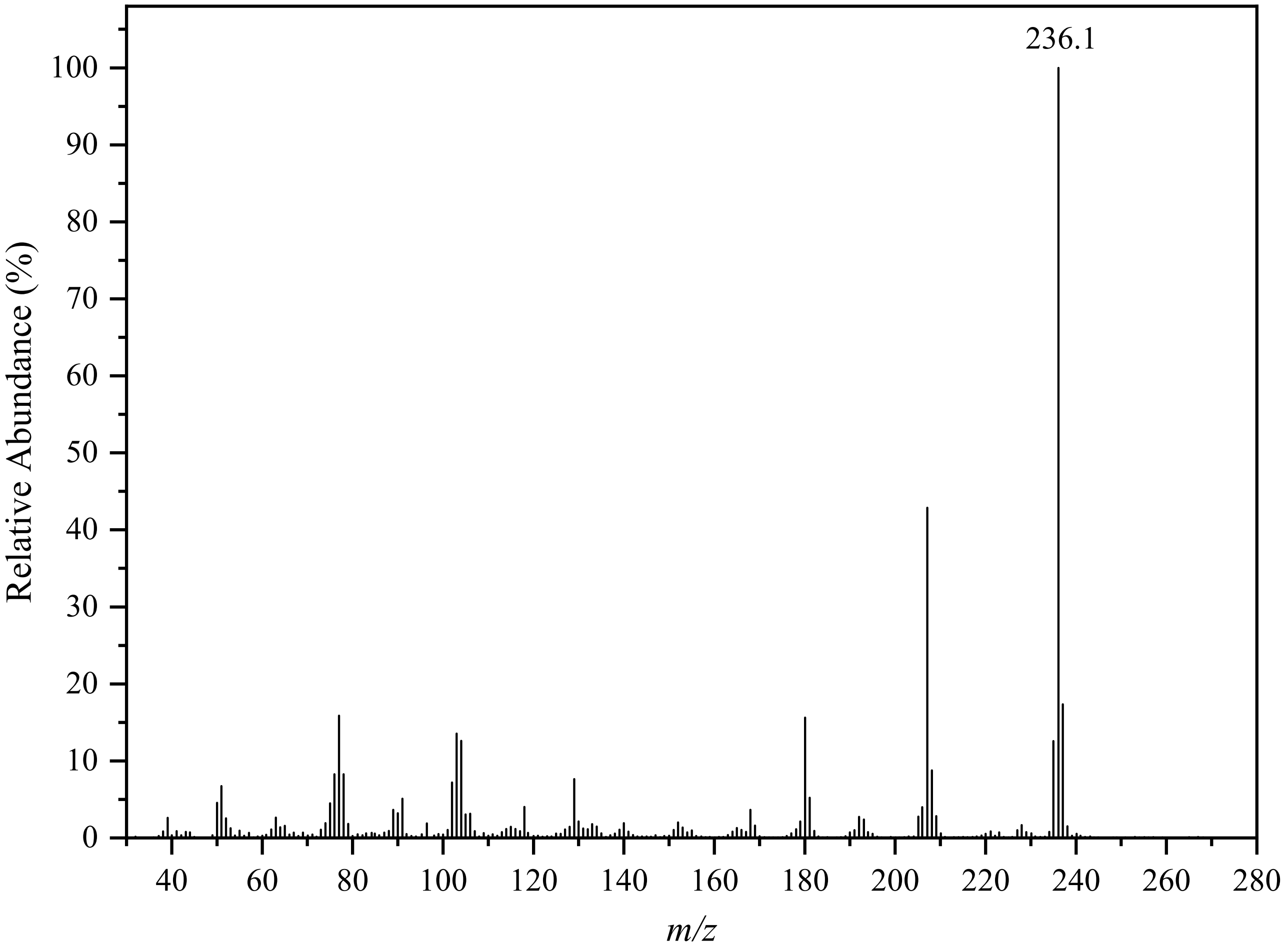


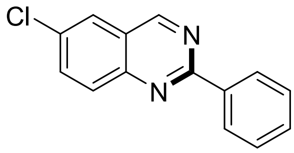


6-methoxy-2-phenylquinazoline (3p)

By following the typical procedure with 2-(aminomethyl)-4-methoxyaniline (0.2 mmol, 30.4 mg), benzaldehyde (0.3 mmol, 31.8 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 80% yield (37.8 mg).

EI-MS: $C_{15}H_{12}N_2O$, m/z (%) = 236.1 (100.0%) [M+].

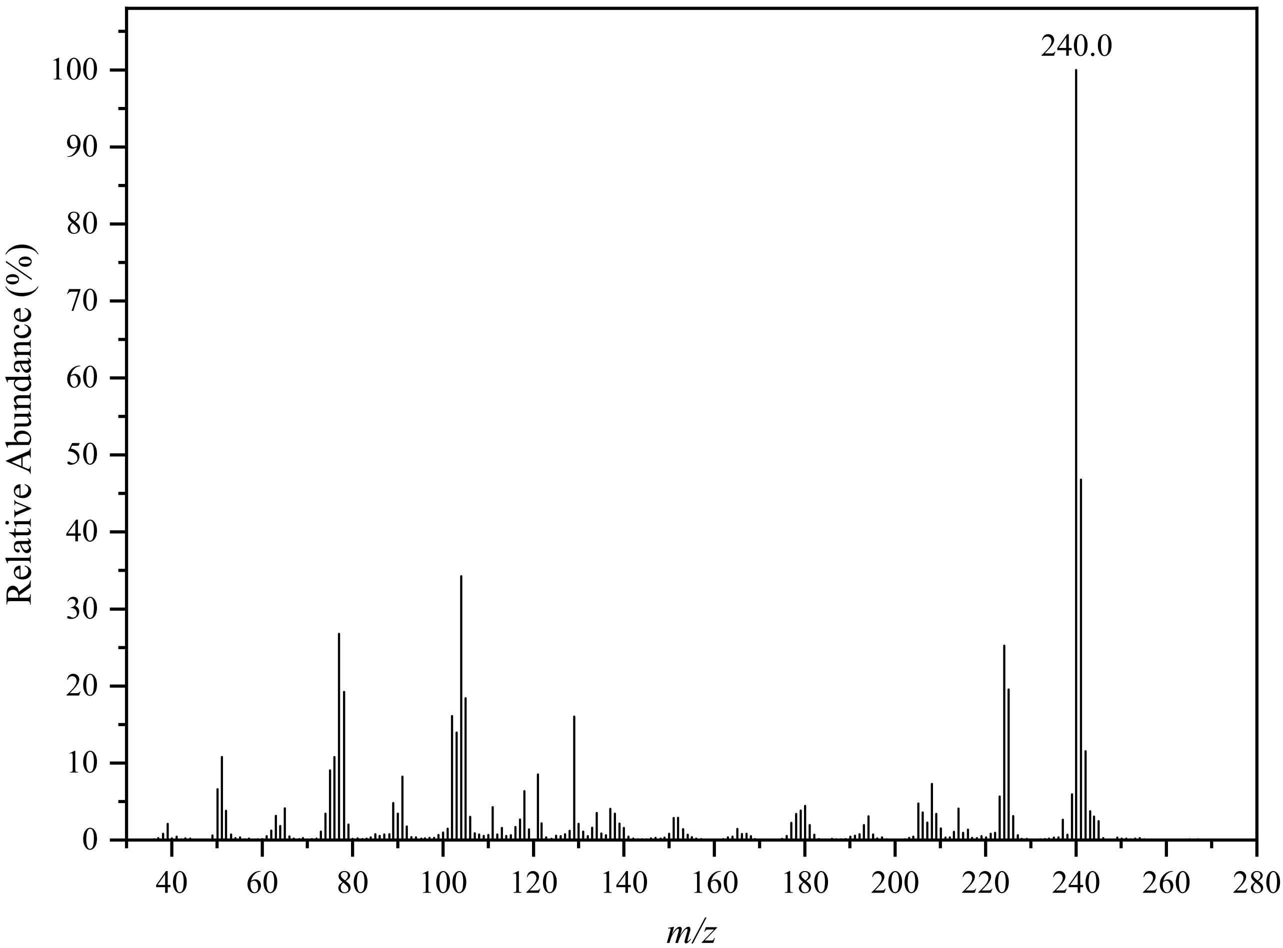




6-chloro-2-phenylquinazoline (3q)

By following the typical procedure with 2-(aminomethyl)-4-chloroaniline (0.2 mmol, 31.2 mg), benzaldehyde (0.3 mmol, 31.8 mg), **$Ce_8As_4W_{36}$** (0.2 mol%, 4.3 mg), the product was isolated with 83% yield (39.8 mg).

EI-MS: $C_{14}H_9ClN_2$, m/z (%) = 240.0 (100.0%) [M+].



5. Notes and References

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