Supplementary Information (SI)

Optimization of Internal Standard Selection for Precise Trace Impurity Quantification in Li_2S via ICP-OES

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Table S1. Mean signal intensities and RSD (%) of each IS from eight replicate measurements (n = 8) in a 1% (w/v) Li_2S matrix spiked with 1 mg/L IS stock solution.

Internal Standard	Line (nm)	Mean Intensity	RSD (%)
Y	371.029	297754.8	1.9
	324.227	25245.8	2.7
Ru	240.272	2448.2	2.0
	349.894	14246.5	1.6
Bi	223.061	1466.5	2.3
	190.171	246.4	1.7
	306.766	52683.7	0.9
In	230.606	799.2	4.0
	325.609	7632.9	2.0
Sc	361.383	97883.9	2.4
	424.683	160718.5	2.5

IS stock solutions (1 mg/L) were spiked into 1% (w/v) Li_2S matrix and analyzed in eight replicates (n = 8). All candidates exhibited RSD values below the 5.0% acceptance threshold, confirming reproducible analytical conditions.

Table S2. Mean signal intensities and RSD (%) of IS candidates measured in DW, 0.1% (w/v) Li_2S, and 1% (w/v) Li_2S matrices from eight replicate measurements (n = 8) at a spiked IS concentration of 1 mg/L.

Interanl Standard	**Line(nm)**	**Deionized**		**0.1% Li_2S**		**1% Li_2S**	
		Ave	RSD(%)	Ave	RSD(%)	Ave	RSD(%)
Y	371.029	423051.6	2.1	391675.2	2.8	334025.2	3.2
	324.227	26268.56	2.9	29786.5	4.9	27601.18	3.7
Ru	240.272	3350.505	4.2	3586.9	6.0	3089.211	3.2
	349.894	11658.73	0.8	16191.3	1.3	18209.45	3.2
Bi	223.061	1649.693	2.9	1823.5	3.0	1699.249	3.2
	190.171	344.9996	1.3	392.0	2.3	315.3068	2.4
	306.766	58344.43	2.6	158073.7	6.3	149818	2.1
In	230.606	2145.933	2.5	1519.3	3.2	1195.567	2.7
	325.609	6754.189	1.6	9254.0	1.1	10396.19	2.8
Sc	361.383	122642.8	2.3	132545.8	3.4	118186.3	3.7
	424.683	215708.4	2.0	239203.7	2.0	208340.6	1.8

Table S3. Normalized relative intensities of IS candidates in 0.1% and 1% (w/v) Li_2S matrices, expressed as the ratio of IS signal intensity in each Li_2S matrix to that in DW. Values close to 1.0 indicate minimal matrix-induced signal perturbation.

Interanl Standard	Line(nm)	0.1% Li2S	1% Li2S
Y	371.029	0.93	0.79
	324.227	1.13	1.05
Ru	240.272	1.07	0.92
	349.894	1.39	1.56
Bi	223.061	1.11	1.03
	190.171	1.14	0.91
	306.766	2.71	2.57
In	230.606	0.71	0.56
	325.609	1.37	1.54
Sc	361.383	1.08	0.96
	424.683	1.11	0.97

Normalized intensities were calculated from mean emission signals of 11 IS candidates (n = 8). Values approaching unity (1.0) indicate minimal matrix-induced signal perturbation; deviations reflect signal suppression or enhancement at each Li_2S concentration.

Table S4. Plasma robustness assessed in DW, 0.1% (w/v) Li_2S, and 1% (w/v) Li_2S matrices using the Mg(II) 280.270 nm / Mg(I) 285.213 nm ionic-to-atomic emission intensity ratio. A Mg(II)/Mg(I) value above 8.0 confirms sufficient plasma excitation temperature for stable ionization under the prevailing matrix load.

Matrix	Mg 285.213(I)	Mg 280.271(II)	Mg ratio(II/I)
0.1 % Li_2S	33277.9	349438.6	10.5
1% Li_2S	30273.7	321166.3	10.6

Plasma robustness was evaluated via the Mg(II) 280.270 nm / Mg(I) 285.213 nm intensity ratio (n = 8) in 0.1% and 1.0% (w/v) Li_2S matrices. Ratios exceeding 8.0 indicate a robust plasma resistant to matrix interferences. The values obtained confirmed adequate plasma conditions at both concentrations, validating the suitability of 1% (w/v) Li_2S for reliable ICP-OES analysis.

Table S5. Recovery (%) of 16 target analytes determined by EC and IS correction using five IS candidates, namely Sc, Y, Ru, In, and Bi, across their respective analytical wavelengths in a 1% (w/v) Li_2S matrix.

Recovery(%) / Elements(nm)	EC	Y	Ru	Bi		Sc	
		324.227	**240.272**	**223.061**	**190.171**	**424.683**	**361.383**
Cu 324.752	65.6	120.5	186.6	143.7	150.1	119.3	117.0
Pb 220.353	45.1	82.8	126.8	**98.0**	**102.1**	81.2	79.7
Zn 213.857	50.9	**93.2**	143.0	110.5	115.3	**91.8**	**90.1**
Cr 267.716	44.8	82.9	127.0	**98.1**	**102.3**	81.4	79.8
Ni 231.604	38.5	74.3	114.1	87.9	**91.7**	72.6	71.3
Al 308.215	62.7	119.8	187.1	143.4	149.9	118.4	116.1
Fe 259.939	47.3	**95.9**	147.1	113.7	118.6	**94.5**	**92.8**
Mn 257.610	43.2	79.2	120.9	**93.6**	**97.6**	77.8	76.3
Ba 455.403	60.7	**107.0**	164.5	127.1	132.6	**105.7**	**103.7**
B 249.772	46.6	84.8	129.9	**100.4**	**104.7**	83.3	81.7
Ca 317.933	61.2	153.9	242.0	185.2	193.9	153.1	150.1
Co 228.616	38.7	72.8	110.8	85.8	89.5	71.3	70.0
Mg 279.077	52.3	**101.0**	154.8	119.7	124.9	**99.6**	**97.7**
Sr 407.771	59.4	**104.5**	160.4	124.0	129.3	**103.1**	**101.1**
Sb 206.836	42.9	83.1	127.8	**98.5**	**102.8**	81.5	80.0
Be 313.107	40.5	72.4	**109.4**	85.1	88.6	71.0	69.8

Analyte recoveries obtained by EC and IS correction were compared for six IS candidates. Standard and IS stock solutions were spiked into 1% (w/v) Li_2S as described in Section 2.2. Recoveries within 100 ± 20% (target: 100 ± 15%) were considered acceptable.

Table S5-1. Comparison of signal intensities and Matrix Effects (ME, %) for six IS candidates in Li_2S matrix and deionized water (DW).

IS nm / IS CPS	Y	Ru	Bi		Sc	
	324.227	240.272	223.061	190.171	424.683	361.383
DW CPS	65126.1	13334.2	4603.4	863.6	441140.2	247296.1
Matrix CPS	35421.2	4644.5	2080.1	373.1	237594.8	136562.9
ME(%)	54	35	45	43	54	55

Table S6. Method detection limits (MDLs) for 16 target analytes determined by external calibration (EC) and internal standard (IS) correction using five IS candidates, namely Sc, Y, Ru, In, and Bi, in a 1% (w/v) Li_2S matrix.

MDL(mg/L) / Elements(nm)	EC	Y	Ru	Bi		Sc	
		324.227	240.272	223.061	190.171	424.683	361.383
Cu 324.752	0.02	0.04	0.08	0.04	0.06	0.04	0.04
Pb 220.353	0.04	0.02	0.05	0.03	0.03	0.03	0.03
Zn 213.857	0.05	0.03	0.01	0.01	0.02	0.03	0.03
Cr 267.716	0.04	0.02	0.01	0.01	0.02	0.02	0.02
Ni 231.604	0.04	0.02	0.02	0.01	0.02	0.02	0.03
Al 308.215	0.1	0.04	0.2	0.08	0.1	0.04	0.05
Fe 259.939	0.04	0.03	0.01	0.01	0.02	0.03	0.03
Mn 257.610	0.03	0.02	0.01	0.01	0.02	0.02	0.02
Ba 455.403	0.05	0.01	0.06	0.03	0.05	0.004	0.004
B 249.772	0.04	0.04	0.02	0.01	0.04	0.04	0.05
Ca 317.933	0.1	0.04	0.18	0.1	0.2	0.04	0.04
Co 228.616	0.03	0.01	0.01	0.01	0.01	0.01	0.01
Mg 279.077	0.05	0.07	0.10	0.06	0.08	0.08	0.08
Sr 407.771	0.06	0.004	0.06	0.03	0.05	0.002	0.003
Sb 206.836	0.04	0.05	0.08	0.05	0.06	0.06	0.06
Be 313.107	0.02	0.02	0.01	0.01	0.02	0.02	0.02

Table S7. LOQs for 16 analytes in a 1% (w/v) Li_2S matrix.

LOQ(mg/L) / Elements(nm)	EC	Y	Ru	Bi		Sc	
		324.227	240.272	223.061	190.171	424.683	361.383
Cu 324.752	0.08	0.1	0.3	0.1	0.2	0.1	0.1
Pb 220.353	0.2	0.07	0.2	0.09	0.1	0.08	0.09
Zn 213.857	0.2	0.1	0.03	0.03	0.08	0.1	0.1
Cr 267.716	0.2	0.07	0.02	0.02	0.05	0.07	0.08
Ni 231.604	0.1	0.07	0.06	0.03	0.06	0.08	0.08
Al 308.215	0.3	0.1	0.5	0.3	0.4	0.1	0.2
Fe 259.939	0.1	0.09	0.03	0.02	0.07	0.09	0.1
Mn 257.610	0.1	0.07	0.04	0.03	0.05	0.08	0.08
Ba 455.403	0.2	0.02	0.2	0.1	0.2	0.01	0.01
B 249.772	0.1	0.1	0.06	0.02	0.1	0.2	0.2
Ca 317.933	0.5	0.1	0.6	0.4	0.5	0.2	0.1
Co 228.616	0.1	0.04	0.04	0.03	0.05	0.05	0.05
Mg 279.077	0.2	0.2	0.3	0.2	0.3	0.3	0.3
Sr 407.771	0.2	0.01	0.20	0.1	0.2	0.01	0.01
Sb 206.836	0.1	0.2	0.3	0.2	0.2	0.2	0.2
Be 313.107	0.08	0.06	0.05	0.04	0.06	0.06	0.07

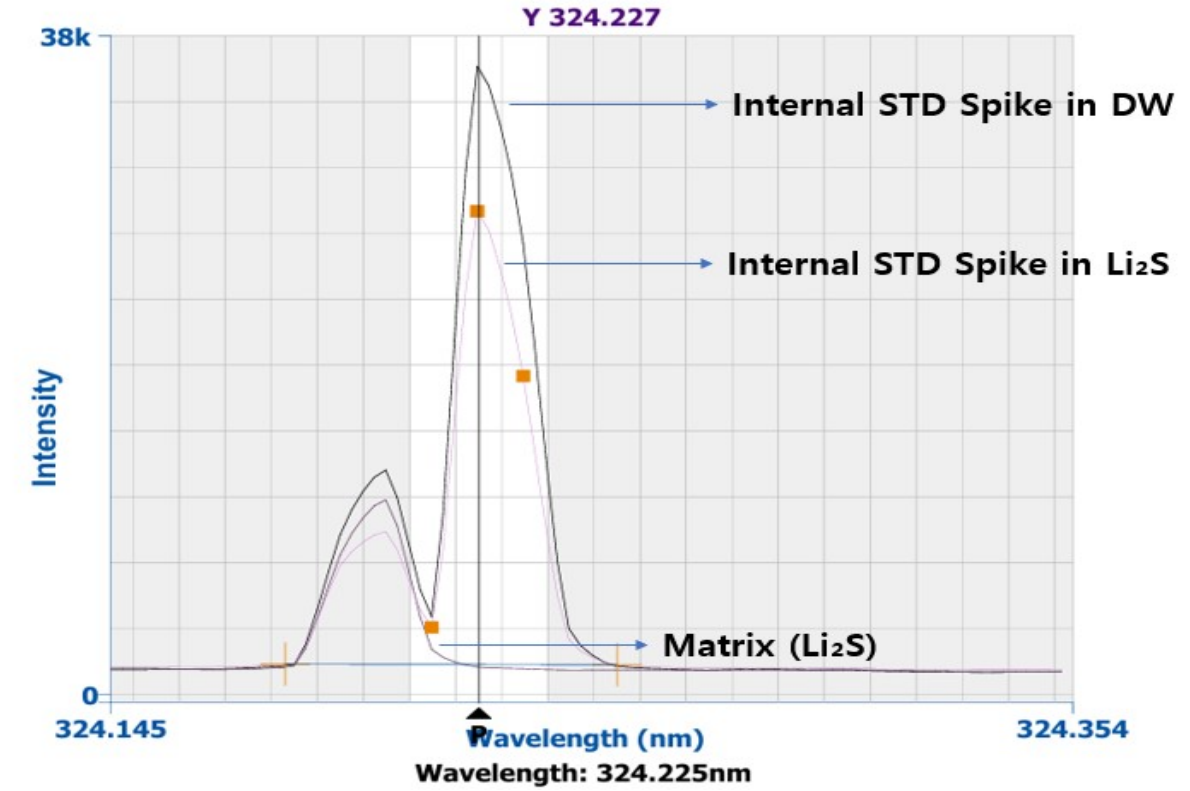
MDL and LOQ were calculated from seven replicates (n = 8) of a 0.25 mg/L standard spiked into 1% (w/v) Li_2S. MDL = 2.998 × s (one-tailed t-value, 99th percentile, 7 d.f.); LOQ = 10 × s, per the US EPA MDL procedure [17].

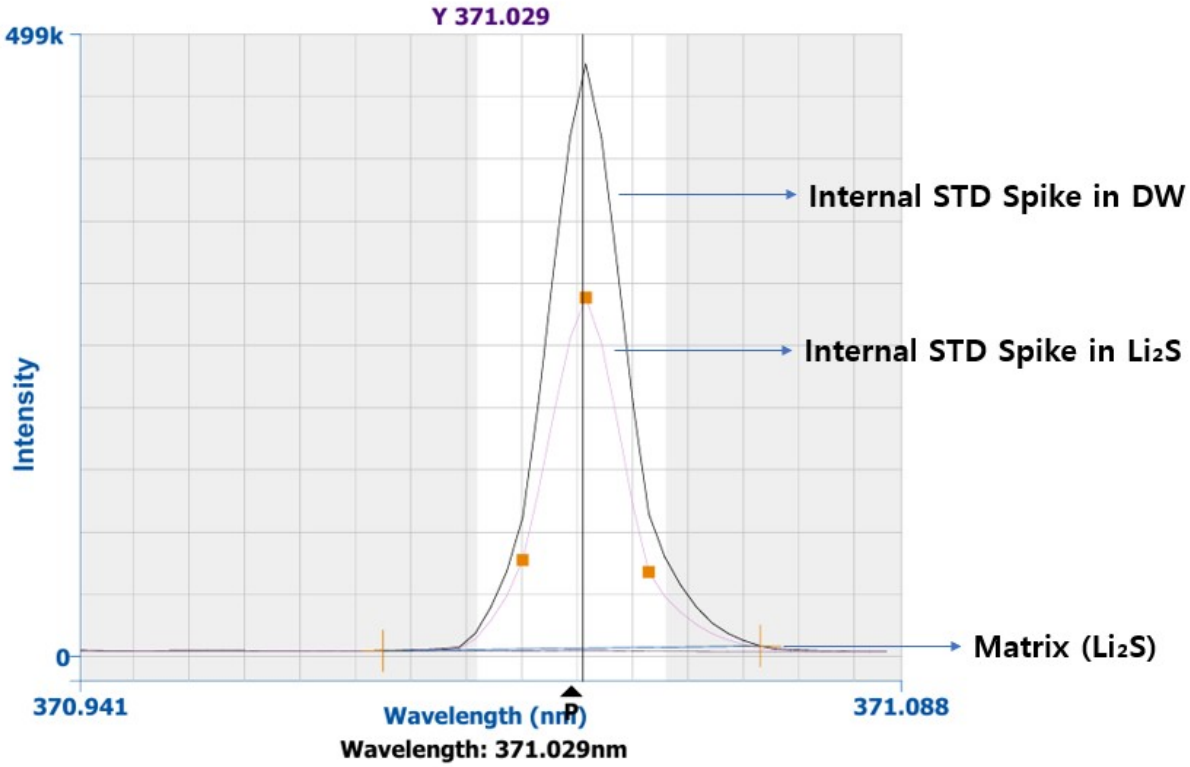
Table S8. Calibration linearity expressed as coefficients of determination (R^2) for 16 target analytes determined by EC and IS correction using five IS candidates, namely Sc, Y, Ru, In, and Bi, across their respective analytical wavelengths.

R^2 / Elements(nm)	Y	Ru	Bi		Sc	
	324.227	240.272	223.061	190.171	424.683	361.383
Cu 324.752	0.999	1.000	1.000	1.000	0.999	0.999
Pb 220.353	0.999	1.000	1.000	1.000	0.999	1.000
Zn 213.857	0.999	1.000	1.000	1.000	1.000	1.000
Cr 267.716	0.999	1.000	1.000	1.000	1.000	1.000
Ni 231.604	1.000	0.999	1.000	0.999	0.999	0.999
Al 308.215	0.999	0.997	0.998	0.997	0.997	0.997
Fe 259.939	0.999	1.000	1.000	1.000	1.000	1.000
Mn 257.610	0.998	1.000	1.000	1.000	1.000	1.000
Ba 455.403	0.999	1.000	1.000	1.000	1.000	1.000
B 249.772	0.999	1.000	1.000	1.000	1.000	1.000
Ca 317.933	0.998	0.995	0.995	0.995	0.993	0.994
Co 228.616	0.999	1.000	1.000	1.000	0.999	1.000
Mg 279.077	0.999	1.000	1.000	1.000	1.000	1.000
Sr 407.771	0.999	1.000	1.000	1.000	1.000	1.000
Sb 206.836	0.999	1.000	1.000	1.000	0.999	1.000
Be 313.107	0.998	0.999	0.999	0.999	0.999	1.000

Calibration curves for all 16 target analytes were constructed in a DW matrix over a concentration range of 0.05–2 mg/L. Both EC and IS correction methods were employed, achieving excellent linearity with $R^2 \geq 0.995$ across all analytes.

Figure S1,S2. Emission spectra of Y (324.227 nm) and Y (371.029 nm) in different matrices.





Emission spectra were compared among IS in deionized water (black), IS in 1% (w/v) Li_2S(pink), and a Li_2S matrix blank (purple) to assess spectral interferences. Both Y lines showed consistent peak positions and shapes regardless of the matrix, with no observable wavelength shifts or overlaps. The Li_2S blank exhibited no detectable signals at these wavelengths, confirming that the discrepancy in normalization effects is not due to spectral interference but stems from energy-dependent matrix effects within the plasma.