



Validation of HPLC analytical method for determination of biogenic amines in agricultural products

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Abstract

An analytical method was validated for the quantitative determination of biogenic amines (BA) in agricultural products. Four agricultural foods, including apple juice, *Juk*, corn oil and peanut butter, were selected as food matrices based on their water and fat contents (i.e., non-fatty liquid, non-fatty solid, fatty liquid and fatty solid), and the precision, recovery, accuracy, limit of detection (LOD) and quantification (LOQ) were determined to test the validity of an HPLC procedure for the determination of BA, including tryptamine, β -phenylethylamine, putrescine, cadaverine, histamine, tyramine, spermidine and spermine, in each matrix. The LODs and LOQs for the BA were within the range of 0.01-0.18 mg/kg and 0.02-0.31 mg/kg, respectively. The relative standard deviation (RSD) of intraday for BA concentrations ranged from 1.86 to 5.95%, whereas, the RSD of interday ranged from 2.08 to 5.96%, respectively. Of the matrices spiked with BA, *Juk* with putrescine at the lowest concentration (10 mg/kg) exhibited the least recovery rate of 89.63%. Therefore, the validation results fulfill AOAC criteria and recommendations. This method has been applying to analyze BA in agricultural products for a total dietary survey in Korea. [This research was supported by a grant (13162MFDS049) from Ministry of Food and Drug Safety in 2013.]

Introduction

4 Matrices

- Apple juice (non-fatty liquid)
- Juk* (non-fatty solid)
- Corn oil (fatty liquid)
- Peanut butter (fatty solid)

Biogenic amines

Limits of biogenic amines for human consumption

In foods:
Histamine: 100 mg/kg
Tyramine: 100-800 mg/kg
 β -phenylethylamine: 30 mg/kg
Total biogenic amines: 1,000 mg/kg

Materials & Methods

Validation

- Precision
- Linearity
- Accuracy
- Recovery

LOD (mg/kg)
LOQ (mg/kg)

Results

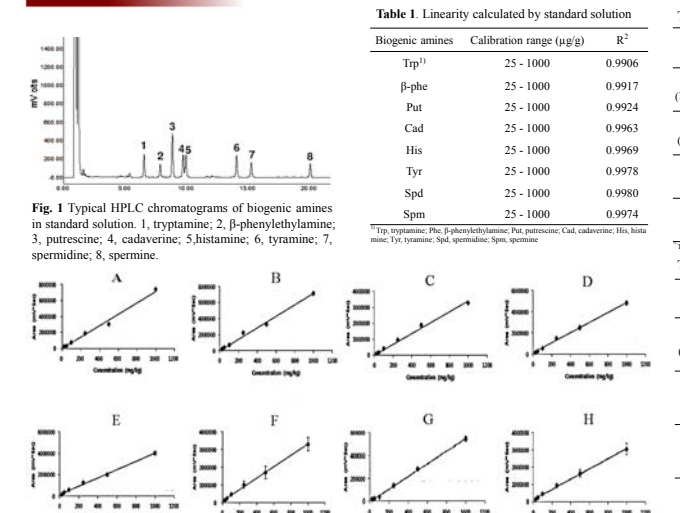


Table 3. Accuracy for the evaluated UV-HPLC methods in samples

Matrix	Concentration ($\mu\text{g/g}$)	Accuracy test (%), n=5										AOAC criteria (%)
		Trp ¹⁾	Phe	Put	Cad	His	Tyr	Spd	Spm			
Apple juice (Non-fatty liquid group)	10	89.85	94.62	101.90	92.42	100.90	94.54	96.83	102.89	80-115		
	100	95.84	87.87	94.20	98.35	91.13	98.67	100.55	101.81	85-110		
	500	93.92	94.83	99.93	98.31	96.85	94.89	92.54	98.31	85-110		
<i>Juk</i> (Non-fatty solid group)	10	108.27	102.36	94.67	109.58	101.89	102.34	109.48	104.44	80-115		
	100	101.05	98.39	109.97	102.11	104.77	101.92	105.95	102.44	85-110		
	500	90.34	90.83	95.87	94.10	97.54	98.12	97.63	87.90	85-110		
Corn oil (Fatty liquid group)	10	94.05	85.97	90.49	86.57	93.88	84.85	86.60	92.79	80-115		
	100	90.34	90.83	95.87	94.10	97.54	98.12	97.63	87.90	85-110		
	500	93.05	94.83	99.13	94.41	96.14	100.23	91.83	100.97	85-110		
Peanut butter (Fatty solid group)	10	93.70	98.27	99.71	99.66	97.55	99.77	93.85	90.29	80-115		
	100	96.32	91.83	96.24	102.24	100.60	97.86	103.71	91.83	85-110		
	500	101.75	93.32	97.60	103.70	101.77	91.19	102.06	103.34	85-110		

Table 4. Precision for the evaluated UV-HPLC methods in samples

Matrix	Concentration ($\mu\text{g/g}$)	Precision (RSD, %), n=5										AOAC criteria (%)						
		Intra day					Inter day											
Apple juice (Non-fatty liquid group)	10	4.58	5.44	3.57	2.63	5.90	4.20	4.19	3.99	4.22	5.44	3.57	4.57	5.15	5.41	4.15	5.40	<6
	100	3.66	3.80	3.35	3.59	2.39	2.82	2.52	2.84	3.92	3.80	3.35	3.68	3.27	3.22	3.94	3.54	<4
	500	3.84	2.08	2.57	3.73	3.46	2.45	3.32	3.00	2.78	2.08	2.57	3.84	3.14	2.68	3.35	3.39	<4
<i>Juk</i> (Non-fatty solid group)	10	3.70	5.21	5.03	4.29	5.06	5.95	3.37	3.01	4.61	3.24	5.03	2.47	5.06	5.95	3.37	4.96	<6
	100	3.69	3.59	3.28	2.48	3.65	3.45	3.18	2.58	3.40	3.59	3.28	2.39	3.65	3.45	3.18	3.50	<4
	500	2.75	3.49	2.12	3.63	3.65	3.90	2.85	3.81	2.23	3.49	3.78	3.73	3.65	3.90	2.85	3.63	<4
Corn oil (Fatty liquid group)	10	4.80	2.58	2.89	2.74	2.31	1.86	4.09	2.22	4.18	3.11	3.23	3.78	3.32	3.06	4.09	4.51	<6
	100	2.82	3.17	3.56	2.73	3.24	2.79	2.16	2.32	2.90	3.17	3.58	2.73	3.29	2.79	2.16	2.32	<4
	500	2.70	2.73	2.25	2.40	1.88	2.19	3.17	2.20	2.90	2.73	2.37	2.40	3.19	2.64	3.17	2.75	<4
Peanut butter (Fatty solid group)	10	4.66	5.41	3.87	4.84	3.15	2.19	4.55	3.61	5.47	5.90	3.68	3.38	3.11	5.96	4.59	5.31	<6
	100	2.56	3.53	2.11	3.48	2.88	2.48	2.85	3.71	3.36	3.14	3.17	3.48	2.88	2.44	3.11	3.94	<4
	500	3.89	3.34	2.97	3.02	3.28	3.20	3.29	2.47	3.02	3.34	2.61	3.02	3.28	3.29	3.75	3.55	<4

Table 5. Recovery for the evaluated UV-HPLC methods in samples

Matrix	Concentration ($\mu\text{g/g}$)	Recovery test (%), n=5										AOAC criteria (%)
		Trp ¹⁾	Phe	Put	Cad	His	Tyr	Spd	Spm			
Apple juice (Non-fatty liquid group)	10	94.78	96.21	103.62	93.98	102.60	96.13	98.46	104.62	80-115		
	100	100.73	92.35	99.01	103.37	95.78	103.70	105.68	107.01	85-110		
	500	97.49	98.44	103.73	102.05	100.54	98.50	96.06	102.05	85-110		
<i>Juk</i> (Non-fatty solid group)	10	102.50	96.90	89.63	103.75	96.46	96.89	103.65	98.87	80-115		
	100	97.61	95.04	106.23	98.64	101.21	98.45	102.35	98.96	85-110		
	500	95.50	97.66	102.18	103.20	99.55	97.23	100.45	96.39	85-110		
Corn oil (Fatty liquid group)	10	106.72	97.55	102.68	98.28	106.53	96.28	98.27	105.29	80-115		
	100	97.12	97.66	103.07	97.17	104.87	95.81	104.97	94.51	85-110		
	500	96.49	98.33	102.79	97.89	99.68	103.93	95.22	104.69	85-110		
Peanut butter (Fatty solid group)	10	97.91	102.68	104.19	104.14	101.93	93.90	98.07	94.35	80-115		
	100	99.80	95.15	99.73	105.94	104.24	101.41	107.46	95.16	85-110		
	500	105.71	96.96	101.41	107.74	105.73	94.74	106.04	107.37	85-110		

Conclusion

- An analytical method was validated for the quantitative determination of BA in agricultural products.
- The validation results fulfill AOAC criteria and recommendations.
- This method has been applying to analyze BA in agricultural products for a total dietary survey in Korea.

References

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