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# Introduction

Color is often the first quality by which cosmetic products are judged. Because compounds used as color additives and their concentration limits are subject to regulations in different countries [1], there is a growing need for analytical control of colorants to ensure that banned additives are not present in cosmetic products and to

determine those permitted by regulations [2]. In this paper, a rapid and sensitive ultra high performance liquid chromatography-tandem mass spectrometry (UHPLC-MS/MS) method utilizing transitions from double-charged precursor ions for measuring ten colorants in lipstick samples was developed.

# Experimental

# Compound informations and the compound-dependent mass spectrometric parameters

Table 1. MRM parameters

No.	compound	Molecular weight	Precursor ion	Product ion	Q <sub>1</sub> Pre Bias (V)	CE (V)	Q <sub>3</sub> Pre Bias (V)
1	Solvent Green 7	524.39	227.90	187.95*	11.0	14.0	19.0
				228.00	11.0	5.0	23.0
2	Acid Red 27	604.47	267.90	228.20*	19.0	14.0	23.0
				268.00	19.0	6.0	29.0
3	Carmine	492.39	267.90	205.90*	19.0	12.0	20.0
				268.05	19.0	5.0	18.0
4	Food Yellow 3	452.37	202.90	206.00*	22.0	25.0	22.0
				203.00	22.0	8.0	13.0
5	Naphthol Yellow S	358.19	312.90	232.95*	22.0	21.0	24.0
				295.90	22.0	24.0	29.0
6	Allura Red AC	496.42	225.00	206.95*	16.0	14.0	21.0
				224.95	16.0	5.0	23.0
7	Ponceau SX	480.42	217.00	199.10*	15.0	17.0	20.0
				217.05	15.0	6.0	22.0
8	Acid Red 87	647.89	646.50	522.65*	32.0	30.0	36.0
				520.70	32.0	25.0	36.0
9	Orange I	350.32	326.70	247.05*	11.0	19.0	26.0
				171.10	11.0	20.0	30.0
10	Acid Orange 7	350.32	326.90	171.05*	16.0	26.0	30.0
10				156.15	16.0	29.0	28.0

<sup>\*</sup> For quantitive use.



### Analytical conditions

HPLC

Mobile phase : A=0.02 mol/L ammonium acetate aqueous solvent; B=Methanol

Flow rate : 0.5mL/min

Column : Shim-pack XR-ODS II 3.0 mm  $\times$  75 mm column (2.2  $\mu$ m)

Oven temperature : 40  $^{\circ}$ C Injection Volume : 10  $\mu$ L

#### MS/MS

Desolvation line temperature: 250 °C
Heating block temperature: 2400 °C
Drying gas: 15 L/min
Nebulizer gas: 3.0 L/min
Dwell time: 40 ms
Pause time: 3 ms



Shimadzu LCMS-8040

# Results

Table 2. Method evaluation and the real sample results

No.	Linear range (mg/L)	Correlation Coefficient	LOD (mg/L)	LOQ (mg/L)	RSD (%)*		Recovery**	RSD***
					RT	area	(%)	(%)
1	0.2-10.0	0.9986	0.0247	0.0987	0.65	2.55	97.3	4.1
2	0.2-10.0	0.9974	0.0424	0.1695	0.12	3.95	63.1	3.2
3	0.1-10.0	0.9983	0.0111	0.0444	0.36	2.88	89.5	2.9
4	0.2-5.0	0.9966	0.0464	0.1854	0.59	1.60	108.0	3.5
5	0.2-10.0	0.9948	0.0061	0.0243	0.83	3.02	117.2	4.2
6	0.2-10.0	0.9991	0.0292	0.1169	0.71	2.33	101.3	3.0
7	0.2-5.0	0.9960	0.0272	0.1089	0.56	3.91	88.5	2.9
8	0.2-10.0	0.9963	0.0206	0.0824	0.79	3.13	97.3	2.2
9	0.2-10.0	0.9960	0.0133	0.0533	0.44	2.02	81.2	1.8
10	0.1-10.0	0.9954	0.0088	0.0352	0.24	4.23	111.0	2.2

<sup>\*</sup>Spiked concentration is 0.5 mg/kg \*\* Spiked concentration is 0.2 mg/kg; \*\*\* RSD for the calculated concentration



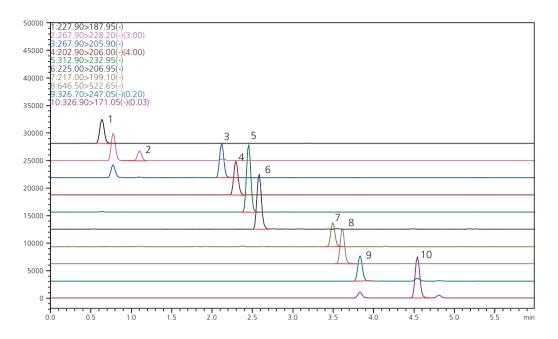


Figure 1. Chromatograms of lipstick samples spiked at 0.2 mg/kg.

### Conclusion

A rapid and sensitive analysis method was developed for the determination of ten colorants in lipstick samples by using UHPLC-MS/MS device. According to the results, the proposed method is ideally suited for the routine monitoring and rapid screening of prohibited colorants in cosmetic products.

# Reference

- [1] González, M.; Gallego, M.; Valcárcel, M. Analytical chemistry. 2003, 75(3): 685-693.
- [2] THE EUROPEAN PARLIAMENT AND THE COUNCIL OF THE EUROPEAN UNION. REGULATION (EC) No 1223/2009 OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 30 November 2009 on cosmetic products[S]. 2009.



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