

ANALYSIS OF HEAVY METALS PRESENT IN LIPSTICK

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Abstract : The study was aimed at assessing the levels of some toxic metals in different Lipstick products sold at different shopping malls and markets in Goa Metropolis. The cosmetic items included eighteen lipsticks of various prices. The were digested and analyzed for heavy metals (lead, cadmium and Nickel) using atomic absorption spectrophotometer. It is obvious from the present study that the use of these lipsticks exposes users to low concentrations of toxic heavy metals which could constitute potential health risk to users since they are known to accumulate in biological systems over time.

KEYWORDS : Estimation of Lead, Estimation of Cadmium, Estimation of Nickel, TGA, UV-Visible, IR, Fluorescence Spectra.

Introduction: Cosmetic is defined as "Any article intended to be rubbed, poured, sprinkled or sprayed on, or introduced into, or otherwise applied to, the human body or any part thereof for cleansing, beautifying, promoting attractiveness, or altering the appearance, and includes any article The present study was conducted to determine heavy metals - lead, cadmium, chromium and nickel in intended for use as a lipsticks.

Materials and Methods

Sampling 1.

16 samples from different brands made from different countries were randomly purchased for analysis but a single brand may contain several colors which have been analyzed separately. Besides, the presence of heavy metals across different colours of the same brand were also sought to be detected and

analyzed. For this, Lipsticks of the commonly used colours pink, red, brown, purple and maroon shades were chosen for studies. They are very popular and most of them are imported mainly from developing countries where no quality control measures are applied.

Methodology : Bees wax - 14gm, white soft paraffin 6gm, olive oil 6ml, pigment 0.7gm, lemon juice 1ml, vitamine E 1ml, vanilla essence 1ml, perfume few ml.

Estimation of lead All chemicals used were of analytical reagent grade or the highest purity available. Doubly distilled de-ionized water, which is non-absorbent under ultraviolet radiation, was used throughout. Glass vassals were cleaned by soaking in acidified solutions of KMnO_4 or $\text{K}_2\text{Cr}_2\text{O}_7$, followed by washing with concentrated HNO_3 and

rinsed several times with de-ionized water. More rigorous contamination control was used when one lead levels in the specimens were low. Cetyltrimethylammonium bromide (CTAB) solution 0.3 M. A 500 ml of CTAB solution was prepared by dissolving 54.67 g of pure cetyltrimethylammonium bromide (E. Merck, Darmstadt, Germany) in 250–300 ml of doubly distilled de-ionized water, sonicated for 30 min and diluted up to the mark with de-ionized water when it became transparent. 1,5-Diphenylthiocarbazone (Dithizone) 1.95×10^{-4} M. Prepared by dissolving the requisite amount (0.005%) of diphenylthiocarbazone (Merck, Darmstadt) in a known volume of 2-propanol (Fluka, Germany). More dilute solutions of the reagent were prepared as required. Lead(II) standard solutions (4.83×10^{-3} M). A 100 ml stock solution (1 mg ml^{-1}) of divalent lead was prepared by dissolving 159.9 mg of lead nitrate (E. Merck, Germany) in de-ionized water. Aliquots of this solution were standardized with EDTA using xylenol orange as an indicator. More dilute standard solutions were prepared from this stock solution, as and when required. Lead(IV) stock solutions (4.83×10^{-3} M). A 100 ml of lead(IV) stock

solution (1 mg ml^{-1}) was prepared by dissolving 110.3 mg of purified-grade lead(IV) oxide (The British Drug Houses LTD England) in de-ionized water containing 1–2 ml of 50% hydrochloric acid. The lead tetrachloride (PbCl_4) solution is stable at room temperature ($25 \pm 5^\circ\text{C}$). The working standard of lead(IV) was prepared by appropriate dilution of this solution. Sodium azide solution. A 2.5% (w/v) sodium azide solution (E. Merck, Germany) was prepared by dissolving the requisite amount in de-ionized water. Tartrate solution. A 100 ml stock solution of tartrate (0.1% w/v) was prepared by dissolving 100 mg of potassium sodium tartrate tetrahydrate (E. Merck, Darmstadt) in (100 ml) de-ionized water. Aqueous ammonia solution. A 100 ml solution of aqueous ammonia was prepared by diluting 10 ml of concentrated NH_3 (28–30%) ACS grade to 100 ml with de-ionized water. The solution was stored in a polypropylene bottle. Other solutions. Solutions of a large number of inorganic ions and complexing agents were prepared from their Analar grade, or equivalent grade, water-soluble salts. In the case of insoluble substances, a special dissolution method was adopted [22]. A series of standard solutions of a neutral

aqueous solution containing 0.6–600 µg of lead (II) in a 10 ml calibrated flask was mixed with 75–130 fold molar excess of a dithizone solution (preferably 1.5 ml of 1.95×10^{-4} M) and 0.3–2.0 ml (preferably 1.0 ml) of 4×10^{-3} M HCl followed by the addition 3–6 ml (preferably 4 ml) of 0.3 M CTAB. The mixture was diluted to the mark with de-ionized water. The absorbance was measured at 500 nm against a corresponding reagent blank. The lead content in an unknown sample was determined using a concurrently prepared calibration graph.

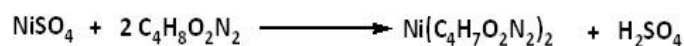
Estimation of cadmium

The calibration curve was plotted for cadmium content in the examined samples ranging from 0.0 to 6.0 µg. For this purpose 0.0, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 5.0 and 6.0 ml of the standard solution of cadmium were measured to separatory funnels and redistilled water was added to 25.0 ml. Further procedure was the same as that used for determination of cadmium. In the basic medium dithizone (diphenylthiocarbazone, H_2Dz) and cadmium (II) ions form pink cadmium dithizonate $Cd(HDz)_2$, soluble in $CHCl_3$ and CCl_4 . The spectrophotometric method of determination of trace amounts of cadmium involves

extraction of the complex of cadmium (II) with dithizonate from the basic medium containing cyanides by means of chloroform, in the presence of sodium-potassium tartrate.

The Gravimetric Estimation of Nickel:

The nickel is precipitated as nickel dimethyl glyoxime by adding alcoholic solution of dimethyl glyoxime $C_4H_6(NO_2)_2$ and then adding a slight excess of aqueous ammonia solution.



When the pH is buffered in the range of 5 to 9, the formation of the red chelate occurs quantitatively in a solution. The chelation reaction occurs due to donation of the electron pairs on the four nitrogen atoms, not by electrons on the oxygen atoms. The reaction is performed in a solution buffered by either an ammonia or citrate buffer to prevent the pH of the solution from falling below 5. If the pH does become too low the equilibrium of the above reaction favors the formation of the nickel (II) ion, causing the dissolution of $Ni(DMG)_2$ back into the mother liquor.

Results of analysis of lead cadmium and nickel

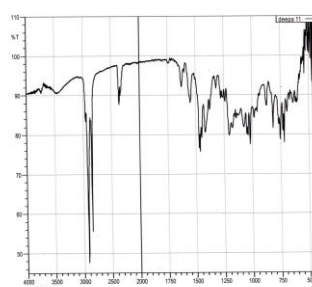
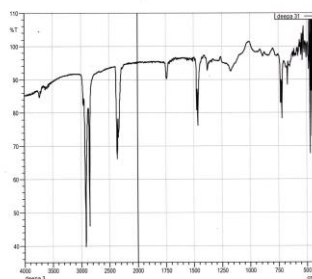
sample number	Lead as Pb	Cadmium as Cd	Nickel as Ni
1.	2.45 mg/kg	0.60 mg/kg	0.0975 mg/kg
2.	1.80 mg/kg	0.30 mg/kg	0.59mg/kg
3.	5.20 mg/kg	0.95 mg/kg	0.95mg/kg

sample number 1-Darzler lipstick

Sample number 2-A.D.S lipstick

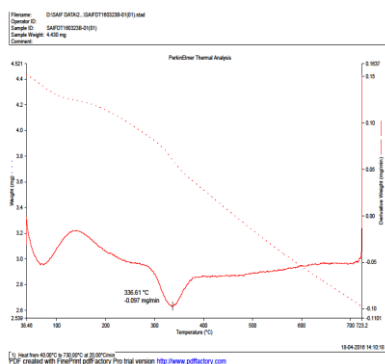
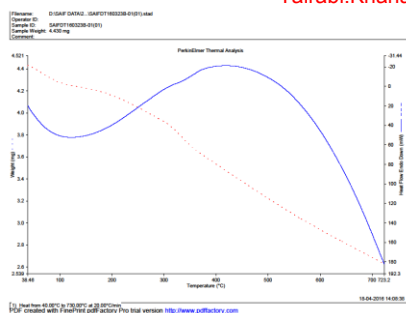
Sample number 3-unbranded lipstick

IR Spectra: Interpretation of the IR spectra of cosmetic products is complicated due to the fact that each of the functional groups visible on the IR spectrum may be responsible for the appearance of multiple bands in a wide range of wave numbers, so that each band may have a contribution of many functional groups.



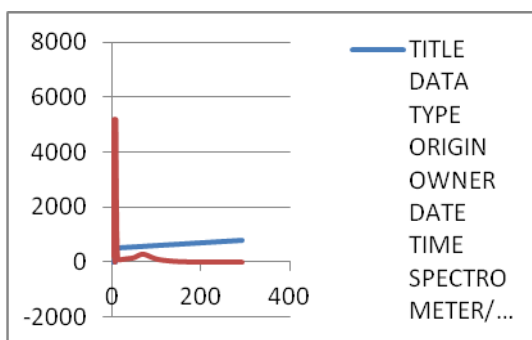
THERMO GRAVIMETRIC ANALYSIS

The technique in which the mass change in a sample is measured as a function of temperature while the sample is subjected to a controlled temperature program. TGA is a branch of material sciences where the properties of materials are studied as they change with temperature. It measures the amount of change in the weight of a material as a function of temperature or the thermal stability of materials and it predicts their thermal stability at temperatures up to 1000°C. This technique can characterize materials that exhibit weight loss or gain due to decomposition or dehydration.



Fluorescence spectroscopy

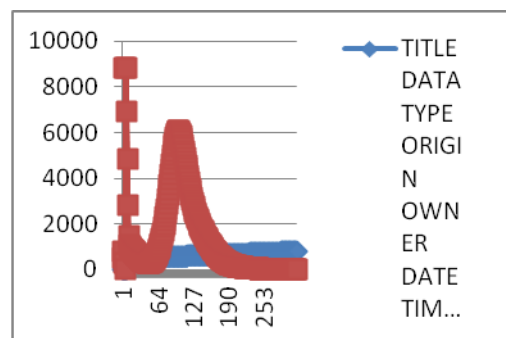
A complementary technique is absorption spectroscopy in the special case of a single molecule fluorescence spectroscopy intensity fluctuations from the emitted light are measured from either single flour pores or pairs of flour pores.



Ultraviolet visible spectroscopy

Refence to absorption spectroscopy or reflectance spectroscopy in the ultra violet visible spectral region this means it uses light in the absorption or

reflectance in the visible range. Directly affects the received color of the chemical involved in this region of the electromagnetic spectrum atoms & molecules undergo electronic transitions from the excited state to the ground state to excited state.



CONCLUSION

The probable causes that may be inferred for the presence of these toxic metals can be stated as following:-

- Contamination at the raw materials stage. Use of substandard raw material in order to save the cost, ignorance created by non technical and under skilled manpower for which there exists no regulation.
- Use of improper grade of water. Faculty water system and both.
- Leaching by the container and the machinery employed.
- Lack of skilled Labor.
- Lack of compliance by small scale cosmetic manufacturers.

- f) Lack of strict regulatory compliance.
- g) Adverse effect cases due to cosmetics use are under reported and poor documentation which gives a free hand to the manufacturers.
- h) Pre market testing is not at all mandatory for cosmetics even by FDA. i) Disjointed legislation at the national and international level.

and Colors. Sec. 74.1306 D&C Red No. 6.

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