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SHORT COMMUNICATION

Quick Semi-quantitative Test for Lead in Processed Foods

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ABSTRACT

A simple, quick semi-quantitative test for the determination of lead metal in canned fruits, vegetables other than dehydrated onion, fish, and meat products, has been developed using the optimised concentration of dithizone reagent under alkaline p*H* as the test solution, which on reaction with the sample solution gives green to blue to violet to red colour, depending on the lead concentration in the sample. The quick test results were compared with atomic absorption spectrophotometric analysis. A total of 80 samples were analysed and it was found that the lead content ranged from 0.001 ppm to 10.35 ppm in all the samples.

Keywords: Lead detection, quick test, dithizone reagent, atomic absorption spectrophotometer, processed foods, lead poisoning, toxicity, canned foods

1. INTRODUCTION

Heavy metals cause health hazards and are toxic to human beings. At certain low concentrations, lead is exceptionally toxic to human beings. Lead exposure may occur due to environmental contamination and also during processing of foods such as solder used in the manufacture of cans is a recognised source of lead contamination in canned foods. Vegetables take up metals by absorbing lead from contaminated soils as well as deposits on parts of the vegetables exposed to the air from polluted environment. Levels of heavy metals including lead in fish have been widely reported[1-5]. The statutory bodies, such as PFA, Bureau of Indian Standards (BIS) who control the quality of foods, have laid down the specifications for lead as maximum of 5 ppm in canned food products, dehydrated vegetables, and 10 ppm in dehydrated onions. Generally, lead content is determined in processed foods by colorimetric procedure but the method is laborious and lengthy. Though atomic absorption spectrophotometer (AAS) is the best choice for accurate analysis, the equipment availability is limited in composite food laboratories because of high cost, however, the ashing step remains the same for both the colorimetry and AAS methods.

Codex[6] has reported the colour test for lead detection. However, simple methodology for lead determination still needs to be developed. Defence Forces are the major consumers of processed foods and the composite food laboratories involved in checking the quality of food supplies do not have easy access to AAS. The modus operandi calls for a quick clearance of samples. In view of these, an attempt has been made to develop simple, quick, semi-quantitative method for determining lead in processed foods.

2. MATERIALS AND METHOD

All the materials, viz., canned fish, meat, vegetables, fruits as well as dehydrated vegetables, were either

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procured from Mysore local market or received from Field Research Laboratory, Leh; Armed Service Corps (ASC) Depot, Bangalore. All the reagents and chemicals used were of AR grade

2.1 Determination of Lead

The sample (1 g) in a silica dish was directly heated on flame for 15 min, 5 ml of 1 per cent nitric acid was added to silica dish to dissolve the burned sample and transferred to a glass test tube. 1 ml of 0.02 per cent dithizone reagent, 2 ml each of 10 per cent citric acid, 5 per cent potassium cyanide, concentrated ammonia solution, and 20 ml of chloroform were added to the test tube.The contents were shaken and after one minute, stable colour was observed.

In a separate glass test tube, known concentration of pure lead nitrate solution (0 ppm to 10 ppm) was taken as standard and 5 ml of 1 per cent nitric acid, 1 ml of 0.02 per cent dithizone, 2 ml each of 10 per cent citric acid, 5 per cent potassium cyanide, concentrated ammonia solution and 20 ml of chloroform were added to the test tube. The contents were shaken and after one minute, stable colour was observed.

The colour obtained by the sample was compared with the colour of known concentration of standard lead solution. Thus, the procedure was standardised for quantification of lead (0 ppm–10 ppm). The colour of the test solution changed from green to blue to bluish-violet to violet to red, depending on the concentration of lead. The samples were also analysed for lead content by M/s Schimadzu Atomic Absorption Spectrophotometer (Model-Anatytik Jenavario 6). Data were statistically analysed using Microsoft Excel.

3. RESULTS AND DISCUSSION

Results of the quick test for lead using standard lead solution are presented in Table 1. The colour of the test solution varied depending on the concentration of lead, and it recorded green (0 to 1.91± 0.08 ppm), blue (2 to 3.91± 0.08 ppm), bluish-violet (4 to 4.91±0.08 ppm), violet (5 to 9.91±0.08 ppm), red (10 ppm and above). Violet was the cutoff point for all the products, except dehydrated onions. These samples were given the numerical score of 1 to 5 for ease in statistical evaluation.

Table 1. Colour of test solution by quick test method with standard lead solution (n=35)

Conc. of lead (ppm)	Colour of sample solution	Score
(0 to 1.91± 0.08)	Green	5
(2 to 3.91± 0.08)	Blue	4
(4 to 4.91± 0.08)	Bluish violet	3
(5 to 1.91± 0.08)	Violet	2
10 and above	Red	1

A large number of samples with varied product profiles, were analysed for lead content both by quick test and AAS methods. The average values of varied brands of the same sample have been tabulated, and in case of single sample, average of duplicate analysis is given. The results have been presented in Table 2.

Table 2. Lead content data by AAS and quick test methods for the various samples (n=80)

Product	No. of samples	Lead by AAS method (ppm)	Colour by Quick test method	Lead by Quick test method (ppm)
Canned Tuna	6	0.157	Green	< 2
Canned Mackerel	5	0.125	Green	< 2
Canned Sardin	4	0.111	Green	< 2
Canned Sardin	1	10.35	Red	> 5
Canned meat and meat products	20	0.234	Green	< 2
Canned fruits and vegetables	33	0.116	Green	< 2
Dehydrated vegetables	10	0.102	Green	< 2
Dehydrated cabbage	1	1.80	Blue	2

Out of the 16 canned fish samples, all except one, showed green colour with the quick test method indicating < 2.0 ppm lead, while by AAS method, lead content ranged from 0.048 ppm to 0.304 ppm. One sample showed red colour with 10.35 ppm by AAS method. Thus, the change in colour reveals the concentration of lead. The formation of green colour complex passes the product as acceptable while red colour complex formation indicate higher level of lead in the product, which is above the specified limit, and thus makes it unacceptable.

Similar kind of data for fish has been reported by other researchers using AAS and inductively-coupled plasma spectrophotometer (ICP) methods[7,8]. Voegborlo[5], *et al*. reported 0.18 ppm to 0.4 ppm of lead in canned fish. Lead content of canned meat and meat products varied from 0.00 ppm to 0.295 ppm by AAS method and the green colour obtained for all the samples by quick test method reflected very low concentration of lead in meat. Vos[9], *et al*., Jorhem[10], *et al*., Aranhas[11], *et al*. and Doganoc[12], have reported lead in meat and meat products as less than the specified limits as determined by AAS method. Thirty-three samples of canned vegetables and fruits, and 10 dehydrated vegetables also showed green colour with the quick test method, indicating < 2.0 ppm of lead. By AAS method, lead content ranged from 0 ppm to 0.261 ppm in these samples Smigiel[13] reported 0.05 ppm to 0.5 ppm of lead in different vegetables. Bahemuka and Mubofu[14] have also reported an average of 0.3 ppm lead in green vegetables. Thus, comparable results were obtained by both the methods, indicating success of the quick test method. The statistical analyses of the data (Table 3), revealed that in canned fish and meat products; and vegetables, the correlation between the quick test and AAS methods was significant, (at 1 %).

Table 3. Statistical analysis of the lead content data for various samples

	Mean (ppm)	Std. Dev.	*r*	r^2
Canned fish	1.060	± 2.910	0.99**	0.98
Canned meat and meat products	0.234	± 0.442	0.98**	0.96
Canned fruits and vegetables	0.116	± 0.313	0.97**	0.94
Dehydrated vegetables	0.189	± 0.566	0.99**	0.98

** significant at 1 % level.

4. CONCLUSION

In the present study, quick test and AAS methods have been compared. However, the merits and demerits of both the methods reflect that the quick test method is a spot test requiring less laboratory facility, semi-quantitative, with 20-25 min analysis time as against 6-7 h by the AAS and colorimetric methods. This method can be used for screening large number of samples quickly and easily.

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