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Research Article

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Investigation of the Alternative Green Procedure for Lassaigne's Test (Quantitative focus)

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Abstract

The research looked into an investigation of the alternative green procedure for Lassaigne's Test with focus on quantitative analysis of Cl- and N. Thus the conventional and the alternative green methods for Lassaigne's Test were compared quantitatively using the significance test of means. The results showed that the qualitative analysis of the Lassaigne's fusion extracts (FE) for Cl^- and N from both the conventional and the alternative green methods were all positive. It was observed that the levels of Cl- were; 34.86 ± 8.74 mg for Na FE, 33.68± 8.12 mg for Zn dust FE, and 32.50 ± 9.76 mg for Al dust FE. The % nitrogen contents that were present in the Na FE, Zn dust FE and Al dust FE were 14.65 ± 0.09%, 14.13 ± 0.22 % and 14.28 ± 0.25 %, respectively. The significance of the means of the results from the methods (conventional and the alternative green method) were determined by using statistical constant (t). The results analyses implied that there is no significant difference between the conventional and the alternative green methods. Thus, due to the accident prone issues and relatively higher cost of Na, it is recommended that the alternative procedures should be totally upheld, deployed and popularized to forestall accident of fire and other injuries.

Keywords: *Lassaigne; Green; Quantitative; statistical constant (t)*

Introduction

Green chemistry is "the utilization of a set of principle that reduces or eliminates the use or generation of hazardous substances in the design, manufacture, and application of chemical products" [1] [2]. There is a need to change or modify the conventional methods which are; not eco-friendly, utilize hazardous solvents, not atom economic. This is useful for the safe being. US EPA describes less hazardous chemicals as substances that are less toxic to organisms, less damaging to ecosystems, not persistent or bioaccumulative in organisms or the environment, and inherently safer to handle and use because they are not flammable or explosive [2]. More so, green chemistry principles call for inherently safer chemistry to prevent accidents. Just imagine that in 2012, in the US alone, there were an estimated 27,500 toxic chemical spills associated with 1000 deaths. UNEP's Global Chemicals Outlook notes that petrochemical accidents in China in 2006 caused losses of approximately USD$11billion – not including injuries, loss or damage to human life, or environmental damage. An explosion at a single plant in France in 2001 caused 30 deaths, 10,000 injuries, and caused damage costs of approximately USD$1.8 billion. Chemical accidents, like the release of methyl isocyanate in 1984 at a factory in Bhopal, India that killed more than 3,800 people, help foster a negative public perception of chemistry. Therefore, Green chemistry seeks to prevent such tragedies by reducing hazards and toxicity [3]. Clearly, the green chemistry principle of inherently safer chemistry has an important role to play in accident prevention [2]. In fact it is believed that the growth of green chemistry can lead to gains in terms of profits, public perceptions of chemistry as whole, and protecting our planet [3].

Couples of efforts have been made thus far in line with green chemistry procedures in order to have less negative print on the environment. For example; preparation of acetanilide, synthesis of dibenzalpropanone, preparation of benzilic acid, Diels-Alder reaction and bromination of trans-stilbene [1]. In addition, alternative green procedure for organic qualitative analysis - sodium fusion test has also been developed. Conventionally, metallic sodium is fused with organic compound (containing some heteroatom). This results into formation of sodium salts. Being soluble in water, the qualitative analysis of the atoms of the organic compound can then be determined. The disadvantages of this conventional procedure for organic qualitative analysis are; it is quite hazardous, hence is often of great concern for students and their safety, and may lead to fire accidents [1]. For the alternative greener procedure, organic sample is carefully mixed with Zn dust and Na2CO3 or Al dust [1]. Furthermore, Green Chemistry is being incorporated at college/university level. It is also being taught to practicing chemists and business leaders [4].

That means the ideas of a green chemistry should become part of their training from the very start. Students of chemistry programs at university should be guided to develop deep consciousness of the importance of sustainability strategies in chemistry research and industry, and also to develop knowledge and skills to operate them [5][3]. We currently have a 'Sustainable Laboratories' programme, working with undergraduate students to substitute hazardous and non-sustainable chemicals used in their laboratory protocols [6]. Green chemistry is spreading from academic labs into industry as a way to reduce costs, as well as environmental, health and safety risks. It also aids in tracking energy use during production, search for sustainable raw materials, and build biodegradable or recyclable products to prevent waste [7]. Hence we report an investigation of the alternative green procedure for Lassaigne's Test (Quantitative focus).

Materials and Methods

Apparatus used includes; fusion tube, test tube, evaporating dish, gauze ,tongs , crucible, desiccators, Kjeldahl flask, heating mantle, clamp holder, wire gauze, tripod stand, stand, measuring cylinder, funnel, filter paper, pestle, watch clock, burette, Erlenmeyer flasks, pH Meter, Pipette, beakers, measuring cylinder, volumetric flask, test tube, weighing machine, conical flasks, dropping pipette, spatula, petri dish, glass funnel, litmus paper.

Reagents/Materials

Reagents and materials used include; sodium hydroxide, ferrous sulfate, ferric chloride, sulfuric acid, acetic acid, nitric acid, sodium nitroprusside, lead acetate, silver nitrate, copper sulphate, sodium sulphate, distilled water, boric acid, methyl red indicator, hydrochloric acid, potassium chromate indicator solution, sodium metal, zinc dust, sodium bicarbonate, filter paper.

Methods

Preparation of Fusion Extracts

About 0.2 g of the organic compound (methyl red indicator or 4-chloro benzaldehyde) was put into the fusion tube followed by 0.3 g of sodium. The tube was held with a pair of tong onto a burner flame. The heating was gently done to avoid spurting out of the sodium. After the sodium was melted, the heating was made strong until the tube's tip became red hot for 5 minutes. Then the hot tube was plunged into 150 mL of distilled water in an evaporating dish and covered with gauze. The tube was crushed with pestle and the mixture was boiled for 5 minutes. The mixture cooled and filtered into a clean Erlenmeyer flask. Thus, colourless and clear filtrates obtained were taken as the fusion extract (Na FE) and were kept for onward qualitative and quantitative analyses. The fusion extracts for the green alternative procedure were similarly obtained using zinc and aluminium dusts - Zn dust FE and Al dust FE - instead of the Na metal [8].

Nitrogen analysis

Standard cyanide test for qualitative analysis of nitrogen was adopted as previously reported [8]. Nitrogen was quantitatively determined using Kjeldahl method [9].

Chloride (Cl^-) determination

Standard test for Cl^- detection with $AgNO_3$ and NH_4OH was used [8], whereas the standard titrimetric determination for Cl- was carried out for the quantitative evaluation [10].

Comparative analysis of the procedures

The comparison of the procedures was carried out using significance of the means analysis. By this, the results from the procedures were determined using statistical constant (t). Calculated statistical constant (tcal) was evaluated as tcal = $|(\bar{X}_{1-\mu})|$□($\sqrt{N}$/SD); where $\bar{X}$ = mean of the new procedure, μ = mean of the conventional procedure, N = number of observation, SD = standard deviation of the new procedure. This method of significance of the means of the results has been previously reported by Ogugbuaja, [11].

Results and Discussion

Results

Results of the qualitative analysis of N and Cl^-

The detection test for N and Cl^- from the Na FE and the alternative green procedure fusion extracts (Zn dust FE and Al dust FE) all confirmed the presence of N from fusion extracts derived from methyl red indicator. Similarly all the fusion extracts (from conventional and alternative green procedure) derived from 4-chlorobenzaldehyde showed positive test for Cl . After all, the test was carried out on fusion extracts of known composition. The key import of the evaluation is that both conventional fusion extract (Na FE) and the alternative green procedure fusion extracts (Zn dust FE and the Al dust FE) derived from methyl red indicator and the 4-chlorobenzaldehyde behave same for these qualitative analyses.

Results of the quantitative analysis N and Cl^-

The amounts of chloride and N present in Na FE, Zn dust FE and Al dust FE are given in Table 1.

Procedure	Sample	Cl^- (mg/L)	N (%)
Conventional	Na FE	34.86 ± 8.74	14.65 ± 0.09
Alternative Green	Zn dust FE	33.68 ± 8.12	14.13 ± 0.21
Alternative Green	Al dust FE	32.50 ± 9.76	14.28 ± 0.25

Table 1: *The amount of N and Cl^-.*

The amounts of Cl^- and N obtained from the conventional procedure were compared to the ones from alternative green procedures using test of significance of means. Hence, the tcal was evaluated and compared with table statistical constant (ttable) at 95% confidence level appropriately. The results are as summarized in Table 2 below.

Discussion

It was observed that all the fusion filtrates from methyl red indicator using Na, Zn dust and Al dust gave positive test for Nitrogen. Again all the fusion filtrates from 4-chlorobenzaldehyde using Na (conventional procedure), Zn dust and Al dust (alternative green procedures) showed positive test for Cl^-. Furthermore, the amount of chloride present in Na, Zn dust and Al dust fusion extracts were 34.86 ± 8.74 mg, 33.68 ± 8.12 mg, and 32.50 ±9.76 mg, respectively. The values were indeed comparable with the theoretical value of 50 mg. Also the amount of nitrogen present in Na, Zn dust and Al dust fusion extracts were determined as 14.65%, 14.13%, and 14.28%, respectively.

Cl- analysis			
Comparison of procedures	**FE**	t_{cal}	t_{table}
Conventional Vs. Green	Na and Zn dust	0.50	12.71
Conventional Vs. Green	Na and Al dust	1.00	12.71
N analysis			
	FE	t_{cal}	t_{table}
Conventional Vs. Green	Na and Zn dust	4.26	12.71
Conventional Vs. Green	Na and Al dust	2.62	12.71

Table 2: *t_{cal} and t_{table} for common and the alternative green procedures.*

These values compared well with the theoretical % N (from methyl red indicator) of 15.60% (see Table 1). In Table 2 the t_{cal} for the determination of Cl^- of 0.50 (Na FE & Zn dust FE) and 1.00 (Na FE & Al dust FE) were less than the t_{table} value of 12.71 (at 95%. This implied that there is no significant difference between the two methods in terms of quantitative analysis of the Cl^-. Similarly, the tcal for %N determination of 4.26 (Na FE & Zn dust FE) and 2.62 (Na FE & Al dust FE) were all less than t_{table} of 12.71 (95% confidence level). Therefore, there is no significant difference between the conventional and the alternative green procedures for the determinations of N as well. Therefore the developed approach for sodium metal replacement with Zn dust and aluminium dust in the Lassaigne's fusion test is efficient and should be upheld, deployed and poluarised with all seriousness in order to avoid the latent fire and injury accident that would result from the conventional procedure of this test.

Conclusion and Recommendations

The qualitative analysis for Cl^- and N for both the conventional method and the alternative green methods were all positive. It was observed that the levels of Cl^- were; 34.86 ± 8.74 mg for Na FE, 33.68± 8.12 mg for Zn dust FE, and 32.50 ± 9.76 mg for Al dust FE. The % nitrogen contents that were present in the Na FE, Zn dust FE and Al dust FE were 14.65 ± 0.09%, 14.13 ± 0.22 % and 14.28 ± 0.25 %, respectively. The significance of the means of the results from the methods (conventional and the alternative green method) were determined by using statistical constant (t). The results analyses implied that there is no significant difference between the conventional and the alternative green methods. Thus, due to the accident prone issues and relatively higher cost of Na, it is recommended that the alternative procedures should be totally upheld, deployed and popularized to forestall accident of fire and other injuries.

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