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# Green Chemistry Laboratories Movement for Sustainability

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## Abstract

Since the past decade the United Nations has emphasized that education should also be for sustainable development. Hence the chemists have focused on sustainability and have started a Green Chemistry Laboratories Movement. Green chemistry, also called Sustainable Chemistry is an area of Chemistry and Chemical Engineering which focuses on designing and synthesizing products to maximise yield, minimizeand eliminate the consumption as well as generation of hazardous substances. Most undergraduate laboratories largely evade the issues of health hazard, occupational safety and environmental threats and have still not incorporated the green chemical methodologies. Teaching Green Chemistry in the class room and the laboratory should be done simultaneously by making it an essential part of the curriculum. The present study proposes Green Chemistry alternatives in the undergraduate course to replace the pre-existing hazardous experiments in accordance with the12 Green Chemistry principles accepted by the United Nationsand the Organization for Economic Cooperation and Development (OECD). This would lead to the improvement of the IAQ (indoor air quality)and eliminates the risk of explosion by making the laboratories less hazardous. This 'Green Laboratories Movement' will encourage the educators and students to replace conventional laboratory experiments by green technologies for safety and sustainability. Such an approach will help students to develop the concepts of green chemistry, laboratory skills, xenobiotics to save the environment and resist climate change.

**Keywords:** *Green Chemistry; hazard; Qualitative; Organic; Lassaigne test;*

## Introduction

The earth is witnessingadeterioration in environment, decline in non-renewable natural resources leading todisappearing ecosystems.Thus the United Nations for the past decade emphasized that education should be for sustainable development. This can be achieved by incorporating Sustainable and Green Chemistry in education at secondary and higher levels to protect and help the economy, people and the planet by finding imaginative and innovative ways to reduce waste, conserve energy, and discover replacements for harmful substances. Laboratory experiments are an important component in Chemistry education at the undergraduate level to inculcate a better understanding of the subject and skill development. Usually the undergraduate laboratories largely evade issues of health hazard, occupational safety and environmental threats by following the traditional experimental work and exercises.In1998 Paul Anastas and John Warner gave the new concept of 'Green Chemistry' with 12 principles(1). The scope of these green chemistry and engineering principles go beyond apprehensions over hazards from injurious chemicals and include energy conservation and waste reduction, as well as the use of more sustainable or renewable feedstock and safe designing for the product as well its final disposition.

On the basis of the 12 green chemistry principles the International union of pure and applied chemistry (IUPAC) adopted Green chemistry in 2000 and defined it as “the invention, design, and application of chemical products and processes to reduce or to eliminate the use and generation of hazardous substances”,the Environment Protection Agency (EPA) gives the definition, “the use of chemistry for source reduction and prevention of chemical hazard”; and the Organization for Economic Cooperation and Development (OECD) gives the definition, “sustainable chemistry”(2,3).These definitions seem to be different from the way they are expressed, but they lead to the same meaning and goal, i.e. the objectives of green chemistry are to design, manufacture and use of efficient, effective, safe and more environmentally benign chemical products and process leading to the control of environmental pollution. The American Chemical Society sees green chemistry as a field that is open to innovation, new ideas, and revolutionary advances(4,5).The integration of sustainability and green chemistry in the education of future chemists and chemical engineers by a Green Chemistry Laboratory Movement will aid active learning in the undergraduate laboratories using a range of green chemistry methods, with a particular focus on hands-on skill development.The present study focuses on the hazards of the qualitative analysis of organic compounds for an undergraduate laboratory courseand to identify experiments which can be replaced by a greener alternative.

**Principles of Green Chemistry**

1.**Prevention.** It is better to prevent waste than to treat or clean up waste after it has been created.

2.**Atom Economy.** Synthetic methods should be designed to maximize the usage of all starting materials into the final product.

3.**Less Hazardous Chemical Syntheses:**Synthetic methods should be designed to use and generate substances that possess little or no toxicity to human health and the environment.

4.**Designing Safer Chemicals:** Chemical products should be designed to affect their desired function while minimizing their toxicity.

5.**Safer Solvents and Auxiliaries:** The use of auxiliary substances (e.g., solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.

6.**Design for Energy Efficiency:** Energy requirements of chemical processes should be recognized for their environmental and economic impacts and should be minimized. If possible, synthetic methods should be conducted at ambient temperature and pressure.

7.**Use of Renewable Resources:** Use of raw materials and feedstocks that are renewable rather than depleting. Renewable feedstocks are often made from agricultural products or are wastes of other processes; depleting feedstocks are made from fossil fuels (petroleum, natural gas or coal) or are mined.

8.**Reduce Derivatives:** Unnecessary derivatization (use of blocking groups, protection/ deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.

9.**Use catalysts, not stoichiometric reagents:** Minimize wastes by using catalytic reactions. Catalysts are used in small amounts and can carry out a single reaction many times. They are preferable to stoichiometric reagents, which are used in excess and work only once.

10.**Design for Degradation:** Chemical products should be designed so that at the end of their function they break down into innocuous degradation products and do not persist in the environment.

11.**Real-time analysis for Pollution Prevention:** Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.

12.**Inherently Safer Chemistry for Accident Prevention:** Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.

**Methodology:**

**Research Design:**

1. Hazard assessment of the experimental exercises were done for qualitative analysis in organic chemistry.

2. Experimental procedures were gathered from educational journals and laboratory manuals on basis of 12 green chemistry principles to replace some of the pre-existing hazardous experiments.

**Qualitative Analysis - Organic**

Traditionally ten steps are involved in the identification of an organic compound after the separation of mixture of compounds like 1)General appearance 2)ignition test, 3)MP/BP 4)unsaturation test, 5)detection of additional elements, 6)solubility7)identification of functional groups, 8)possible compounds 9)preparation of derivatives and 10) result.Most of these steps involve non-green experiments and alternatives were sought for in the educational journals.

An organic compound is composed of carbon, hydrogen and oxygen but sometimes hetero atoms like Nitrogen(N), Sulphur(S), Halogens(CI, Br, I and F),Phosphorous(P)are also present and aredesignated as special/additional elements(6).The qualitative detection of either the presence or absence of the additional elements acts as a diagnostic tool for characterization of a compound.In 1843, Lassaigne devised a method for detecting nitrogen present in an organic compound by fusing the compound with potassium metal(7).Due to the production of hazardous potassium cyanide Jacobsen replaced potassium with sodium for fusion of organic compound(8,9).The sodium metal is known to be highly reactive and explosive in nature therefore its handling is difficult for it often catches fire. Sodium cyanidegenerated in Lassaigne's test is also very lethal and endangers theenvironment at a slow but constant rate.In 1935, one modification of this test known as Middleton test was proposed where organic compounds were heated with zinc powder and sodium carbonate(10,11,12)to make the fusion extract instead of native Na metal. The Middleton's test transformed Nitrogen, Halides, Phosphate into sodium cyanide, sodium halides, sodium phosphate and zinc sulphide respectively. Zinc sulphide generated was insoluble in water but dissolved in hydrochloric acid while all other sodium salts were water soluble. This differential solubility of zinc sulphide made the identification process convenient. ()

## Experimental

### Traditional Lassaigne's test (Detection of Additional Elements)

The qualitative detection of either the presence or absence of special elements **(N, S, Cl, Br, I)** acts as a diagnostic tool for characterization of a compound. Several procedures were known but the most familiar one was Lassaigne's test.

Preparation Of Sodium Fusion Extract: Sodium metal is fused in an ignition tube by heating it red hot over the flame and plunging in distilled water to convert the water insoluble organically bound additional elements to water-soluble sodium salts which can be easily detected by various tests.

$$Na + C + N \longrightarrow NaCN$$

$$Na + S \longrightarrow Na_2S$$

$$Na + X \longrightarrow NaX$$

where (X = Cl/Br/I)

**Hazard:** Sodium metal is explosive in nature and the Sodium cyanide generated in Lassaigne's test is a lethal chemical.

**Green Non-hazardous and safe procedure**: Use of zinc and sodium carbonate instead of metallic sodium.(13,14,15)
Organic sample (about 10 mg) is thoroughly mixed with a mixture of Zn dust (20 mg) and $Na_2CO_3$ (30 mg) powder in a fusion tube, heated first gently and then strongly in the flame till it becomes red hot and kept insame condition for two minutes. The bottom part of the fusion tube is plunged into 5 ml of distilled water taken in a mortar, ground well with the pestle and filtered. With the filtrate tests for S, N and Cl / Br / I are carried out in the same manner as in the case of Lassaigne's Test.

**Precautions:**

1. The fusion tube must be heated very strongly, keeping at red hot condition for at least two minutes. If not properly heated, fusion is not take place (as in case of sodium also), and thus expected observation (colour change) may not be observed. In that case, it is advised to repeat the fusion.
2. The amount of water taken in the mortar must be within 5 ml.; otherwise, the solution will be too dilute to respond to tests.

3. While carrying out the test for nitrogen, ferrous sulphate crystals are to be added; not the solution. This is to avoid excessive dilution.

4. Acidification must be carried out with dilute $H_2SO_4$, not with HCl.

5. No ferric chloride should be added.

| S. No. | Experiment | Observation | Inference |
|---|---|---|---|
| 1. | 0.5 ml filtrate + $FeSO_4$ crystal heat + dil. $H_2SO_4$ | Prussian blue colour | N present |
| 2. | a) 0.5 ml filtrate + Sodium nitroprusside | Black ppt | S present |
| | b) filtrate + dil acetic acid + Lead acetate | Black ppt | S present |
| 3. | filtrate + $FeCl_3$ | Blood-red ppt | N+S both present |
| 4. | filtrate + 2 drops Conc $HNO_3$ boil cool + $AgNO_3$ | Curdy white ppt (Soluble in $NH_4OH$)<br><br>Pale yellow ppt (Partly soluble in $NH_4OH$)<br><br>Yellow ppt (Insoluble in $NH_4OH$) | Chlorine present<br><br>Bromine present<br><br>Iodine present |

**Result:**

**Green context:**

This experiment totally eliminates the risk of explosion and fire hazard which are often met while carrying out the same experiments using metallic sodium.

Improves the IAQ (indoor air quality) of the lab.

The aforesaid zinc-alkali mixture (prepared by intimately mixing 2 parts by weight of zinc dust and 3 parts by weight of sodium carbonate can be stored in a stoppered bottle for more than a month (14).

**Discussion:**

The qualitative analysis for the identification of the organic compound usually practiced in the undergraduate laboratories has many non-green experiments and one of them being the detection of the additional element called the Lassaigne's test. It was observed that the Na metal used in the Lassaigne's test for making the fusion extract sometimes catches fire and flies out from the ignition tube. It is a potential hazard for first time learners. Both the fusion filtrates one from the conventional method using Na and the other from Zn dust and sodium carbonate gave positive test for Nitrogen, Sulphur and the halogens. Therefore, there is no significant difference between the conventional and the alternative green procedures. The green chemistry approach for sodium metal replacement with Zn dust and sodium carbonate in the Lassaigne's fusion test is efficient and should be upheld, deployed and popularised with all seriousness in order to avoid the latent fire and injury accident that would result from the conventional procedure of this test.
Adhering to the 12 principles of Green Chemistry it is proposed that all the experiments should be conducted in semi-micro or micro-scale. Thin-layer chromatography (TLC), spectroscopic techniques (UV, IR and wherever available NMR) should be the methods of choice for determining purity, functional groups and structure elucidation. Eco-friendly organic solvents like ethanol and methanol should be used as an alternative to ether, petroleum ether and ethyl acetate. Ethyl chloroformate can be substituted for $PCl_5$, $PCl_3$, $POCl_3$ or $SOCl_2$. Dimethyl carbonate may be used as a suitable substitute for dimethyl sulphate and methyl halides for methylation(14). Experimental procedure with longer reaction time and at high temperatures should be avoided.

**Conclusion:**

The laboratory course in qualitative analysis for organic is fraught with hazards and includes quite a few non-green experiments. Rediscovery of a green alternative of Lassaigne's test is the Middleton testin which Na metal was substituted with Zn dust and $NaCO_3$to give identical results which aids in identifying the organic compound. It also eliminates the risk of explosion and fire hazard which are often met while carrying out the same experiments using metallic sodium. It improves the IAQ (indoor air quality) of the lab and makes it a better place to work. The incorporation of this non-hazardous green chemistry alternative will be a step taken towards Green Chemistry Laboratories Movement for sustainability of our planet earth.

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