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Greening Qualitative Organic Analysis: Spot Test to Detect Aromatic Hydrocarbons and Aryl Halides

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Abstract Chemistry students in their college laboratory routinely employ qualitative reactivity patterns to identify functional groups of unknown organic compounds. Qualitative organic analysis is therefore very useful for determining the overall structure. Most of the common functional groups can be easily detected by simple color tests that are rapid, simple and routinely performed by the students. However, the detection of aromatic hydrocarbons and aryl halides is still a challenge as there are not many color tests available. The present work highlights the detection of aromatic hydrocarbons and aryl halides by simple modification of their reaction with chloroform. The method is simple, using chemicals that are easily available in the undergraduate laboratory and follows the principles of green chemistry.

Keywords: *qualitative organic analysis, undergraduate laboratory, aromatic hydrocarbon, aryl halide*

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1. Introduction

Science students in the undergraduate curriculum routinely perform a number of chemical tests in qualitative organic analysis as a part of their chemistry class. This methodology trains the students in various chemical procedures and techniques and makes them more proficient in the organic laboratory. To be a successful chemist, one must have a strong grasp of the foundations of chemistry, extensive laboratory experience, and knowledge of green chemistry concepts. These courses prepare the students for the challenges that come with a job in research and development. Qualitative organic analysis is a combination of various physical and chemical tests that will be used to determine the functional groups present in an organic compound. [1,2] Each of these tests should ideally give an easily visible result. In general, colour tests have the most advantage - in terms of simplicity and convenience of usage. There is no need for expensive equipment or chemicals, and the student takes only a few hours. Depending on the test used, a negative colour test result can serve to rule out the presence of a particular functional group. However, certain drawbacks do exist. These tests are often performed either as macroscale [1,2] or microscale analysis [3] and the process of performing the tests often generates a significant amount of waste which is difficult to dispose of. [4] Furthermore, the improper disposal of chemicals not only comes at a high cost but has negative consequences for the environment. In addition, very often, in many procedures, hazardous chemicals are used.

During the past few years, our department has been focussed on the development of simple spot tests for the easy and rapid identification of various functional groups in our college undergraduate laboratory. [4,5] These tests have allowed the students to routinely identify various functional groups of organic compounds using the principles of green chemistry - without the use of harsh chemicals, avoiding the production of wanted waste/by-products, while saving time and energy. [6] During the past few decades, the practice of green chemistry has gained much importance in chemical laboratories, including the undergraduate labs. [7,8] Green chemistry is a cleaner and safer approach to chemistry, reducing and avoiding the risks and hazards generally associated with the use of chemicals. [9,10,11] Performing simple coloured spot tests is a green way to organic qualitative analysis. [12]

Aromatic hydrocarbons are derivatives of benzene, [13] containing carbon and hydrogen, and relatively unreactive and difficult to identify by simple tests. In the most common test, the aromatic compounds react with chloroform in the presence of anhydrous aluminium chloride to produce coloured triarylmethanes cations, Ar_3C^+. Generally, the colour depends on the number of rings in the particular aromatic hydrocarbon. Usually, benzene and its derivatives tend to give an orange-red colour; while naphthalene and its derivatives give deep blue-purple colours. [1,2] It is important to note that the above test requires the use of anhydrous (water free) aluminium chloride and the students must ensure that the test tube and other materials are clean and dry before performing this test.

2. Experimental

Conventional Procedure: In a clean, dry pyrex tube, heated $AlCl_3$ horizontally and added 4-5 drops of the given aromatic hydrocarbon and 1 mL of $CHCl_3$. Shake and note the colour change. [1,2]

Spot Test: In a clean, dry groove tile, add 40 mg anhydrous $AlCl_3$, 2 drops or 20 mg of the aromatic hydrocarbon and 3-4 drops of chloroform ($CHCl_3$) or dichloromethane (CH_2Cl_2) and mix well.

3. Result and Discussion

In our continuing efforts to develop simple colour based spots for the detection of various functional groups in organic qualitative analysis, a spot test was developed for the detection of aromatic hydrocarbons based upon the conventional method with anhydrous aluminium chloride and chloroform. The test is performed on a groove tile and requires a lesser quantity of the reagents. The test can be performed with either chloroform or dichloromethane (a safer choice in terms of toxicity). Furthermore, the test can be expanded to aryl halides such as chloro- and bromobenzene, where a distinct orange colour is observed.

Table 1. Colour observed with various aromatic hydrocarbons and aryl halides with anhydrous $AlCl_3$ in either chloroform or dichloromethane via the green method

S. No	Compound	Colour with $CHCl_3$	Colour with CH_2Cl_2
1	Naphthalene	violet	purple violet
2	Anthracene	violet	green
3	Phenanthrene	violet	violet
4	Biphenyl	violet	purple
5	Acenaphthene	violet	violet
6	Chlorobenzene	orange	orange
7	bromobenzene	orange red	orange
8	toluene	orange	orange
9	xylene	orange red	orange
10	Acetanilide	yellow orange	yellow orange
11	Nitrobenzene	No significant color	No significant color
12	Phenol	No significant color	No significant color
13	Benzaldehyde	No significant color	No significant color
14	Acetophenone	No significant color	No significant color
15	Phenylacetic acid	No significant color	No significant color
16	Benzoic acid	No significant color	No significant color

3.1. Comparison of Analysis

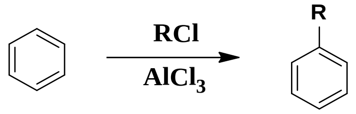

Table 2. Comparative study - Conventional test method vs Spot test

Criterion	Conventional (macro)	Spot test (micro)
Anhydrous $AlCl_3$ (amount required)	200mg	50 mg
Organic Compound (aromatic hydrocarbon or aryl halide)	50 mg	20 mg
$CHCl_3$/CH_2Cl_2	0.5 mL	1-2 drops
Heating	Required	Not required
Time	5-10 minutes	1 minute

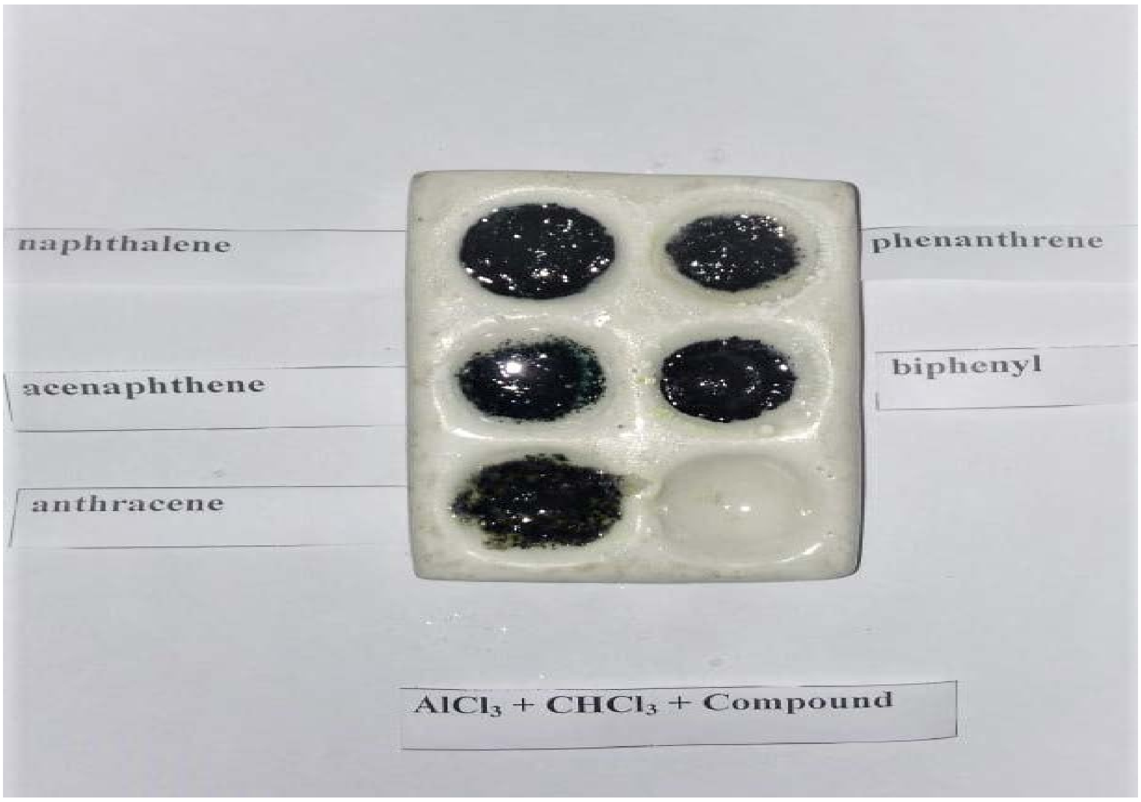

Figure 1. Observed colours via spot test performed on groove tile with aromatic hydrocarbons using anhydrous $AlCl_3$ and chloroform

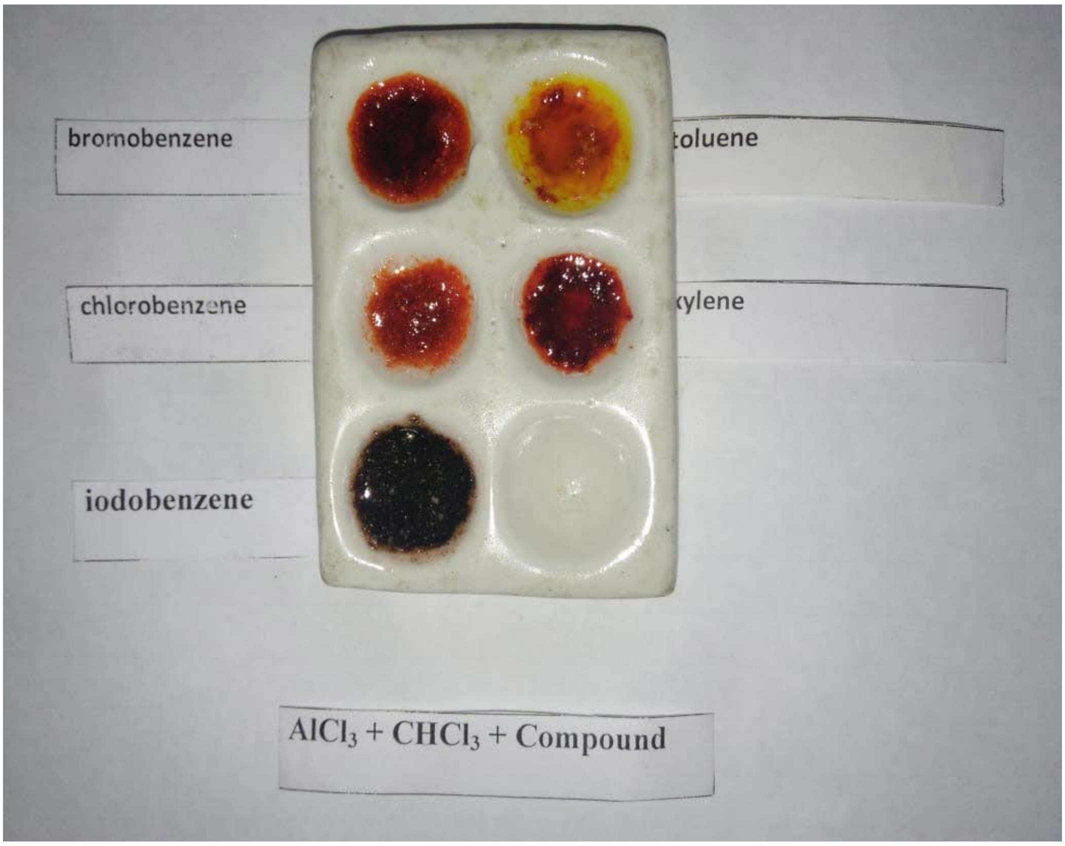

Figure 2. Observed colours via spot test performed on groove tile with aryl halides and aromatic hydrocarbons (toluene and xylene) using anhydrous $AlCl_3$ and chloroform

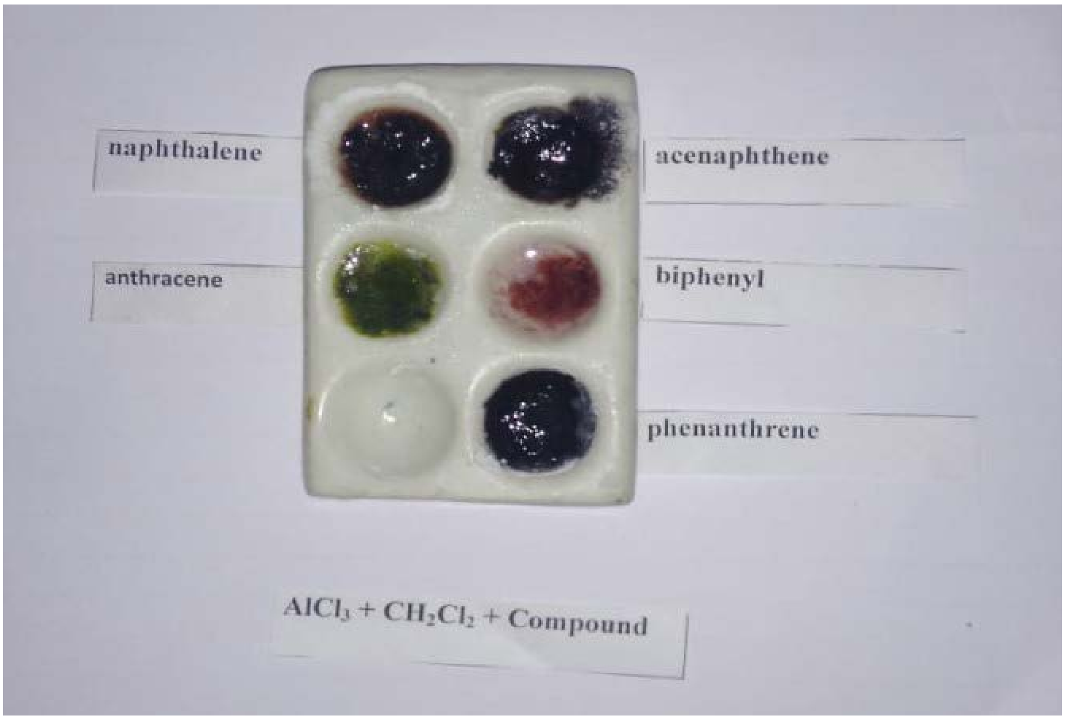

Figure 3. Observed colours via spot test performed on groove tile with aromatic hydrocarbons using anhydrous $AlCl_3$ and dichloromethane

4. Conclusion

A simple and easy to perform spot test is developed to identify aromatic hydrocarbons and aryl halides using a modified Friedel-Crafts reaction with anhydrous aluminium chloride and chloroform. The test is performed on a groove tile, requires less amount of the reagents and colour change is observed in seconds. In terms of green chemistry, the spot test is safe, saves energy and consumption of chemicals is less. The test will be an easy addition to the undergraduate organic analysis procedure.

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