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EDITORIAL

AJCE IS ABOUT TO PUBLISH A SPECIAL ISSUE ON THE EVE OF ACRICE 3

Temechegn Engida
Email: temechegn@gmail.com

Dear AJCE Communities,

The African Conference on Research in Chemistry Education (ACRICE) FASC’s official conference on chemistry education. It takes place every two years rotating among the chemical societies in Africa events. It is an IUPAC recognized conference. ACRICE-3 will take place in Setif/Algeria during the week of 8 October 2017.

Sometime around the event of the ACRICE-3, AJCE has planned to publish its special issue on the theme of SYSTEMIC APPROACH TO TEACHING AND LEARNING CHEMISTRY (SATLC). SATLC was initiated some 20 years back and AJCE wants to celebrate it through publishing a special issue. AJCE therefore invites you to submit manuscripts in this particular theme as soon as possible.

Enjoy the second issue of AJCE 2017 that contains articles on various issues of chemical education.

SJIF IMPACT FACTOR EVALUATION [SJIF 2012 = 3.963]
KNOWLEDGE, ATTITUDES AND PRACTICES ASSOCIATED WITH PESTICIDE USE AMONG HORTICULTURAL FARMERS OF BANJULINDING AND LAMIN OF THE GAMBIA

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ABSTRACT

This work takes a limited but significant look at knowledge, attitudes and practices (KAP) vis-à-vis pesticide use in two farming communities in The Gambia. Most of the users have no formal education and they never received any formal training related to pesticide use. However, for the most part, there was a very high level of awareness among users about the dangers posed by pesticides. In addition, most of the respondents do not respect pesticide labels. More than 50% of the respondents experienced symptoms of pesticide poisoning, and about one-fifth of these sought medical help. Lack of knowledge, unsafe attitudes and dangerous practices were identified as the leading cause of pesticide poisoning. [African Journal of Chemical Education—AJCE 7(2), July 2017]
BACKGROUND

Any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating any pest is called a pesticide [1]. Pesticides are used widely in the agricultural sector to boost productivity by preventing or minimizing the effects of pests on farm produce. Alongside this is, pesticide poisoning in humans. This is prevalent in developing countries.

Lack of knowledge, unsafe attitudes and dangerous practices have been identified as the leading courses of pesticide poisoning. In their joint statement titled “children are facing high risks from pesticide poisoning” in 2004, the FAO, UNEP & WHO pointed out that developing countries use only a quarter of the global pesticide production yet account for 99% of all deaths attributable to pesticides [2]. Most pesticide intoxications may be due to lack of knowledge, unsafe attitudes and dangerous practices. Adherence to instructions on the pesticide is the principal rule in ensuring safe pesticide handling. Once this is deficient, storage, mixing and application of pesticides could result in poor health consequences.

However, it is worth noting that high level of awareness does not always imply positive attitude/practices. For example, in assessing the KAP and toxicity symptoms associated with use and exposure among farm workers in the Gaza strip, Yassin et al [3] found that although the farmers had high knowledge about the adverse health effects of pesticides and many self-reported toxicity symptoms, they however made very poor use of protective measures. This prompted Yassin et al to suggest that it would be useful to encourage the use of alternative measures to conventional pesticides. In Ghana, despite the high level of awareness of the health effects of pesticides [4], the use of personal protective equipment is minimal. This, Clarke [4] noted, was due to financial constraints. Kuye et al [5] found both knowledge and the use of personal protective equipment to be low among cotton farmers in his land research in the Central and Upper River
Regions of The Gambia. On the other hand, in Thailand, found 89.4% of their respondents wore personal protective equipment although majority of them (77.2%) had low level of knowledge.

Illiteracy and limited safety training and practices in face of widespread pesticide use in developing countries especially in small scale farmers is a huge challenge [6]. In a landmark research in The Gambia, Kuye et al [5] pointed out that there is inadequate control of pesticides and other hazardous agrichemicals in The Gambia after concluding that there was a low level of awareness on the toxicity of pesticides. Farmers in developing countries lack training on pesticides. For example, Naido et al, [6] indicated that only 16.4% of sprayers had ever attended a pesticide-training course among study participants in rural KwaZulu-Natal, South Africa.

Pesticides can enter the body by one or all of three routes: ingestion, inhalation or absorption.

Unsafe practices that have been found in developing countries include storing pesticides in family bedrooms (where they could be easily reached by children), use of empty containers for domestic purposes, eating/drinking/smoking while handling pesticides [7]. Individuals that have a long working experience with pesticides have been reported to take fewer preventive measures when working with pesticides [8].

Pesticide poisoning can lead to both acute and chronic illnesses. Many of the short-term health effects of pesticides go unreported unless they are life threatening requiring visit to a health center. Fatigue, headache, body ache, skin discomfort, skin rashes, poor concentration, feelings of weakness, circulatory problems, dizziness, nausea, vomiting, excessive sweating, impaired vision, tremors, panic attacks, watery eyes, sneezing, burning sensation especially around the eyes etc. and in severe cases coma and death have been mentioned as immediate symptoms [9].
AIMS AND OBJECTIVES

The goal of this study is principally to explore the knowledge, attitudes and practices (KAP) of farmers in the Banjulinding Women Horticulture and Lamin Horticulture Farms in the Kombo North District of The Gambia. Comparatively, more women are involved in farming compared to men. Banjulinding has a thriving farming community; this is why it was selected for this study.

The objectives of this study were to explore the knowledge, attitudes and practices (KAP) of farmers as regards the use of pesticides.

1. Are they similar to what were obtained in some parts of the sub-region?
2. How well do farmers protect themselves when using pesticides?
3. How knowledgeable are they about the health effects of pesticides?

The researchers in this study hypothesized that farmers have low level of awareness, negative attitudes and unsafe practices regarding the use of pesticides.

The Pestizid Aktions-Netzwerk e.V. Germany, in their document on pesticides and their health effects pointed out that, pesticides can cause chronic illness if incorporated over long periods of time even in relatively small amounts. Non-Hodgkin lymphomas, cancer of the prostate, pancreas, lungs, ovaries, the breasts, testicles, liver, kidneys and intestines as well as brain tumors, disruptions in cognitive and psychomotor functions, depression and damaged central nervous system have all been associated with pesticide use [9].

RESEARCH METHODOLOGY AND DATA COLLECTION

A descriptive cross sectional study design was used. Sixty-six subjects were randomly selected from two community horticultural farms in the Kombo North District of The Gambia.
(Kombs is a local term that means urban area). Data was obtained from the respondents through the administration of a semi-structured questionnaire. The informed consent of each respondent was obtained prior to the administration of the questionnaire. A descriptive cross-sectional study design was used to explore the KAP of the farmers. Epi Info 7.1.4.0 was used to obtain a representative sample of 66 study subjects.

The study sites are the Banjulinding Women’s Horticulture Farm and Lamin Horticulture Farm. They are located in the villages of Banjulinding and Lamin of the Kombo North District respectively in The West Coast Region of The Gambia. Banjulinding and Lamin are communities characterized by successful community gardens. The Banjulinding Women’s Horticulture Farm is one of the most successful community farms in the country. It was established in the early nineties. The garden was revitalized in 1996 with advent of the Republic of Taiwan. There were 115 workers in the garden. Only 10 of them were men. Lamin Horticulture Farm was established in 2013. It was funded through the Gambia Agricultural Development Lowland Project under the Ministry of Agriculture. The farm employs 96 workers. Ninety-three of them are female.

Data for the study were collected through the administration of a semi-structured questionnaire. The questionnaire was divided into three sections.

1. Section A looked at the demographic information and general KAP of the respondents.
2. Section B assessed the KAP of the respondents towards personal protective equipment.
3. Section C examined their KAP on the health effects of pesticides.

Informed consent of each respondent was verbally granted before the questionnaire was administered. The purpose of the research was also thoroughly explained to respondents. Respondents were also told of their right to withdraw from the interview at any point they deemed
necessary without any repercussion and that their names will not be written on any part of the questionnaire for any reason.

An obvious limitation of this study is that it was not nation-wide. Availability of resources constrained the researchers to limit their zone of study to the two farming communities.

RESULTS

Demographic information

Of the subjects of this study, 54.55% received non-formal education (Arabic), 24.24% received primary education, 10.61% secondary education and the rest (10.60%) were illiterates. Farming is considered as a job for illiterates. Number of years working with pesticides ranged from 1 to 42 years with an average of 11 years. The vast majority (89.39%) of the respondents receive their pesticides labeled. The majority (55.45%) of the respondents did not attend any training related to pesticide use. A quarter (24.24%) of the respondents could not mention any symptom of pesticide poisoning. However, they all knew pesticides could cause ill health or even death. More than 78% of the respondents knew pesticides can enter the body through the nose, mouth and eyes but less than half (48.48%) knew through the skin. A few (10.61%) of the respondents believed their bodies had developed immunity against pesticides. Most (56.06%) of the respondents do not follow pesticide labels/instructions. Some farmers (10.61%) store their pesticides in their homes and the same percentage used buckets to apply pesticides. None of the farmers eats, drinks, smokes or engages in discussion while working with pesticides; however, 1.52% did admit to chewing kola nut while using pesticides. Only 15.15% always used Personal Protective Equipment (PPE) during pesticide storage, 15.15% during mixing and 33.33% during
application. About half (54.55%) have had symptoms after pesticide use but only 20.59% of them ever went to the hospital after experiencing symptoms.

Of the 66 farmers interviewed, females formed the majority accounting for up to 75.76% (n = 50) of the total respondents. The ages of the respondents ranged from 21 years to 78 years with an average of 48 years.

Knowledge

When asked where they obtained their pesticides, 21.21% (n=14) said they obtain theirs from Gambia Horticultural Garden Center and the rest (n=52) obtain theirs from other shops in the market. 89.39% (n=59) of the farmers acknowledge receiving their pesticides usually labeled and the rest (10.61%) said their pesticides are usually unlabeled. 54.55% of the respondents never attended a training related to pesticide use while 45.45% of the reported to have received training related to pesticide use.

When interviewed about the signs or symptoms of pesticide poisoning, 30.30% of the respondents mentioned sneezing, 3.03% diarrhea, 6.06% nausea, 7.58% watery eyes, 21.21% irritation, a quarter (25.76%) mentioned burning sensation around the eyes, 6.06% dizziness, 12.12% vomiting, 18.18% headache and 24.24% of them could not mention any correct sign or symptom of pesticide poisoning. All the respondents knew pesticides could enter the body through the nose, 78.79% knew pesticides can enter the body through the skin, 89.39% knew through the mouth and 78.79% knew through the eyes.

About three-quarters (71.21%, n=47) knew other methods for pest control but only 45.45% (n=30) ever used these methods. All (n=66) of the respondents know that pesticides can cause ill health or death. About two-thirds (65.15%) of the respondents use more than one type of pesticide to control pests on their plots while 34.85 indicated that they only use one type of pesticide to
control pests on their plots. However, 92.31% (n=60) of the respondents could not name any pesticide they use.

**Attitudes**

When the respondents were asked whether they believe pesticides enter the body through the nose, skin, eyes and mouth, 93.94% (n=62) of them said they believe so while the remaining 6.06% (n=4) said they did not believe so. Some of the respondents (10.61%, n=7) believe that their bodies have developed immunity against pesticides over time and 89.39% (n=59) do not believe that their bodies have developed immunity against pesticides over time. All (n=66) of the respondents think they need to take bath after pesticide use. There was an individual who said that he no longer smells pesticides and he attributed it to long time exposure to pesticides and nonuse of protective equipment particularly the nose mask.

**Practices**

About half (56.06%, n=37) of the respondents do not follow pesticide labels (manufacturers prescription) while 43.94% (n=29) of them do not follow pesticide labels. Majority (n=48) of the respondents do receive help from either relatives/coworker (n=8) or someone (n=40) who has received training on pesticides.

![Chart 1: Pesticide Storage](chart.png)
Three-quarters (75.76%, n=50) of the respondents do not go opposite the wind direction when applying pesticides, however 24.24% (n=16) do go opposite the wind direction when applying pesticides. About one-fifth (21.21%, n=14) of the respondents burn their empty pesticide containers, 7.58% (n=5) bury them in the ground, 27.27% (n=18) discard them in bins/dumpsites, 30.30% (n=20) throw them in toilets while the remaining (13.64%, n=9) do other things with them.

Table 1: Actions during pesticide use

<table>
<thead>
<tr>
<th>Action</th>
<th>Yes</th>
<th>No</th>
</tr>
</thead>
<tbody>
<tr>
<td>Smoke</td>
<td>0% (n=0)</td>
<td>100% (n=66)</td>
</tr>
<tr>
<td>Drink</td>
<td>0% (n=0)</td>
<td>100% (n=66)</td>
</tr>
<tr>
<td>Eat</td>
<td>0% (n=0)</td>
<td>100% (n=66)</td>
</tr>
<tr>
<td>Chew e.g. kola or gum</td>
<td>1.52% (n=1)</td>
<td>98.48% (n=65)</td>
</tr>
<tr>
<td>Engage in discussion</td>
<td>0% (n=0)</td>
<td>100% (n=66)</td>
</tr>
</tbody>
</table>

When the respondents were asked whether they take a bath before engaging in other activities after pesticide use, 74.24% (n=49%) of them responded yes while 25.76% (n=17) said
no. Fifty-five (83.33%) of the respondents take at least 24 hours to re-enter their plots to work and 16.67% (n=11) of them take less than 24 hours to re-enter their plots to work after they have been sprayed.

**Personal Protective Equipment (PPE) use**

All (n=66) the respondents acknowledged that PPE use is important to them because they (PPE) offer protection against pesticide entry into the body.

*Table 2: PPE use among the farmers*

<table>
<thead>
<tr>
<th>Activity</th>
<th>Always (n=66)</th>
<th>Sometimes (n=66)</th>
<th>Never (n=66)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Storage</td>
<td>15.15% (n=10)</td>
<td>22.73% (n=15)</td>
<td>62.12% (n=41)</td>
</tr>
<tr>
<td>Mixing</td>
<td>15.15% (n=10)</td>
<td>28.79% (n=19)</td>
<td>56.06% (n=37)</td>
</tr>
<tr>
<td>Application</td>
<td>33.33% (n=22)</td>
<td>33.33% (n=22)</td>
<td>33.33% (n=22)</td>
</tr>
</tbody>
</table>

*Table 3: PPE use frequency*

<table>
<thead>
<tr>
<th>PPE</th>
<th>Yes (n=66)</th>
<th>No (n=66)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gloves</td>
<td>56.06%</td>
<td>43.94%</td>
</tr>
<tr>
<td>Goggles</td>
<td>31.82%</td>
<td>68.18%</td>
</tr>
<tr>
<td>Hat</td>
<td>15.15%</td>
<td>84.85%</td>
</tr>
<tr>
<td>Face shield</td>
<td>28.79%</td>
<td>71.21%</td>
</tr>
<tr>
<td>Special boots</td>
<td>31.82%</td>
<td>68.18%</td>
</tr>
<tr>
<td>Overall</td>
<td>43.94%</td>
<td>56.06%</td>
</tr>
<tr>
<td>Nose mask</td>
<td>57.58%</td>
<td>42.42%</td>
</tr>
</tbody>
</table>

**Experience on the health effects of pesticides**

Out of the 66 respondents, 34 (51.52%) have had symptoms after pesticide use. The table below shows the symptoms that were reported and the number of farmers that reported each.
DISCUSSIONS

Demography

From the analysis, most of the respondents were married (the rest widowed) and more than three quarters (75.76%) of them are women. This difference in gender is acceptable considering the common Gambian believe that horticultural gardening is more of a feminine activity. This finding is in line with Murphy’s [10] research in Indonesia that highlighted that majority of her respondents were women.
Knowledge

Although most (about 71%) of the respondents know other methods for pest control, their use is very low because the farmers believe they are not as effective as chemical pesticides. This finding is different from that of Abang et al’s [11] findings in the tropical region of Cameroun. They alluded that the low level of knowledge on other pesticide control measures was due to the farmer’s lack of access to information on integrated pest management. Methods for pest control the respondents mentioned include the use of a mixture of soap and neem leaves and the use of ash. They pointed out that they received training on these methods although they hardly use them.

Practices

Of the 46 people that received help during pesticide use, majority (82.61%) did so from someone who had received specialized training on pesticide use. The trained helpers mostly only do the application process. During the application process, the farmers’ role is to help in pulling the pipe (when a motorized sprayer is used) as the pesticide is being applied on the crops.

Pesticide storage was not a problem for the vast majority of the farmers. However, in conformity with Abang et al’s [11] study some of them do store their pesticides in completely unacceptable places - homes. The farmers that store their pesticides at home said they feel their pesticides are safer at home. Even though most of the farmers use appropriate equipment to apply pesticides, some of them use very inappropriate equipment such as buckets and cans. Those using inappropriate equipment do so because they believe that they can apply the pesticide where they exactly want it and will not get into the financial trouble of buying tin milk for another person to apply pesticides for them. About a quarter of the farmers do go opposite the wind direction when applying pesticides and their reason was that that it prevents the pesticides from escaping into the air.
Most of the farmer’s practices about empty pesticide containers are poor. Similarly, Naidoo et al [6] reported that women working in small-scale agriculture in South Africa mostly burn, bury or just throw away empty pesticide containers; and these poor practices were associated with limited training on pesticide use. The farmers have a very good practice of not eating, drinking, smoking, chewing or engaging in discussion during pesticide use.

**Personal Protective Equipment (PPE) use**

There is high level of awareness about the importance of PPE. However, its use is very low among the respondents and even the few that do use them are always inadequately dressed during pesticide use as reported by Kuye et al [5] among cotton farmers in the Central River and Upper River Regions of The Gambia. In this research, the principal reason for not using PPE was unavailability.

**Experiences on the health effects of pesticides**

Similar to Clarke et al’s [4] findings, a good proportion of the respondents reported having had symptoms after working with pesticides. The high proportion of respondents having symptoms could be linked to the very low use of PPE and inappropriate attitudes. Most of the people that had symptoms only drank milk and felt that they were okay. However, some of them reported having gone to the hospital after the symptoms became severe.

**CONCLUSIONS AND RECOMMENDATIONS**

There is appreciably high level of awareness among the people covered by this study about the harm that pesticides can do. However, knowledge and practices seem to be far apart; the users do not practice what they know. There is a flagrant disregard for caution. Personal protective
equipment use is very low among the farmers and their practices after experiencing pesticide symptoms are worrying; for the most part, they do not seek medical help.

Knowledge regarding pesticide use especially on the potential of causing health problems is high. The attitudes of the farmers have also been at a greater extent positive. However, the farmer’s practices during pesticide use have not always been guided by their knowledge and attitudes. Personal Protective Equipment use is very low among the farmers and their practices after experiencing pesticide symptoms are worrying.

From the findings of this research, the following recommendations are hereby made:

1. The responsible department/agency should ensure regular training of farmers.

2. The responsible department/agency should institute and enforce occupational health and safety measures that will protect farmers.

3. There is a need for more research on agricultural pesticide use in The Gambia.

REFERENCES


APPENDIX - QUESTIONNAIRE
Section A: Demographic information and General KAP

Demographic information
1. Sex: □ Male □ Female
2. How old are you? .........................
3. Marital status: □ Single □ Married □ Divorced
4. What is your educational status?
   □ Primary □ Secondary □ Tertiary □ Non formal □ Others ......................
5. How long have you been using pesticides? ...........................................

Knowledge
1. Where do you obtain your pesticides? ..................................................
2. Are they labeled? □ Always □ Sometimes □ Never
3. Have you ever attended a training related to pesticide use? □ Yes □ No
4. Name any signs or symptoms of pesticide poisoning you know. ..................
5. Name the routes of pesticide entry into the body you know.
   □ Nose □ Skin □ Mouth □ Eyes
6. Do you know other ways for pest control rather than chemical pesticides use?
   □ Yes □ No
7. If yes, do you use them? □ Yes □ No
8. Do you know pesticides can cause ill health or even death? □ Yes □ No
9. Do you use one type of pesticide to control all pests on your farm? □ Yes □ No
10. Can you name the pesticides you use? ...................................................
11. Which pest do you use it to control? A. Ants B. Flies C. Both D. Others ..............

Attitudes
1. Do you believe pesticides enter the body through the nose, skin, eyes and mouth?
   □ Yes □ No
2. As a farm-worker, do you believe that your body has developed immunity against pesticides over the time? □ Yes □ No
3. Do you think you need to take bath after pesticide use? □ Yes □ No

Practices
1. Do you follow pesticide labels/manufacturers prescription? □ Yes □ No
2. Do you receive help during pesticide use? □ Yes □ No
3. If yes, from who?
   □ Relative/co-worker □ Expert (someone who has received training on pesticides)
5. What do you use to apply pesticides?
   A. Bucket B Knapsack/motorized sprayer C Watering can D Others specify
6. Do you go opposite the wind direction when applying pesticides? ☐ Yes ☐ No
7. What do you do with empty pesticide containers?
   A. Burnt B. Wash and carry home C. Discard in bin/dumpsite D. Other Specify…
8. During pesticide use, do you do the following?
   i. Smoke ☐ Yes ☐ No
   ii. Drink ☐ Yes ☐ No
   iii. Eat ☐ Yes ☐ No
   iv. Chew e.g. kola nut or Gum ☐ Yes ☐ No
   v. Engage in discussion ☐ Yes ☐ No
9. Do you take bath before engaging in other activities after pesticide use? ☐ Yes ☐ No
10. After applying pesticides, how long does it take you to re-enter the plot to work?
    ☐ <24hrs ☐ >24hrs

Section B: Personal Protective Equipment (PPE) usage
1. Is wearing PPE important to you? ☐ Yes ☐ No
2. If yes, why? ………………………………………………………………………………………………
3. Do you use personal protective equipment during the following?
   i. Storage ☐ Yes ☐ sometimes ☐ No
   ii. Mixing ☐ Yes ☐ sometimes ☐ No
   iii. Application ☐ Yes ☐ sometimes ☐ No
4. Which of the following PPE do you use?
   i. Gloves ☐ Yes ☐ No
   ii. Goggles ☐ Yes ☐ No
   iii. Wide brimmed hat ☐ Yes ☐ No
   iv. face shield ☐ Yes ☐ No
   v. Special boots ☐ Yes ☐ No
   vi. Overall ☐ Yes ☐ No
   vii. Nose mask ☐ Yes ☐ No

Section C: Experiences on the health effects of pesticides
1. Have you had any symptoms after pesticide use? ☐ Yes ☐ No
2. If yes, tick the symptoms. Sneeze, watery eyes, irritation, diarrhea, nausea, dizziness, burning sensation especially around the eyes, others ………………………………………

Thank you very much for accepting to participate in this study!
INSTRUCTIONAL QUALITY OF LOWER GRADES NATURAL SCIENCE CLASSES: THE CASE OF PRIMARY SCHOOLS LINKED TO KEMISE COLLEGE OF TEACHER EDUCATION

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ABSTRACT
The main aim of this study was exploring the quality of natural science education classroom instruction in lower grade primary schools linked to KCTE. The study also sought to assess the extent to which classroom instructions is helping in filling the gap of students` science learning. The study employed mixed method within the descriptive survey research design. Of 14 primary schools linked to KCTE, eight of them were selected by using convenient sampling. Grade three, four and six sections were selected using probability sampling using lottery method. Grade three, four and six science teachers in the selected schools and eight schools` principals were selected using availability sampling. Questionnaire, interview and observations including recording were used to collect data. The collected data were analyzed using both qualitative and quantitative techniques. The finding of the study showed that the science classroom instructions have gaps in providing professional knowledge, practices and in exploiting methods of integrating science subjects. Based on the findings it is recommend that natural science teachers need capacity building training on pedagogical and subject matter issue, closer support and supervision. It is also recommended that Natural Science Department of KCTE should offer workshops on practical science teaching-learning and on selected topics of science content for the primary schools science teachers and school science department heads. Principals and cluster supervisors should work together to support the teachers and to make close supervision of the classroom instructions. Science teachers in schools need to engage themselves in professional dialogue. [African Journal of Chemical Education—AJCE 7(2), July 2017]
INTRODUCTION

Science is a human attempt to understand the mysteries of the natural world around us through research disciplines and ways of thinking rationally. It involves using the knowledge that has been generated through process skills to create and constantly refine testable models of nature that help us to describe, explain, predict and to conceptualize observable phenomena of nature; through processes such as observations, questions rising and hypothesing that ideas are used in trying to explain new evidence. Consequently, science aims to develop the conceptual and procedural understanding among the pupils, which were not developed independently, and to develop scientific process skills, to foster the acquisition of concepts and to develop particular attitudes. Further, science allows students to explore their world and discover new things and an important part of the foundation for education for all children [1]. That is why while learning science, children`s ideas as starting points, practical investigation, the local environment, balance between knowledge and skills, integration and linkage principle incorporated were important. The owing to facilitate a process of active construction, context-specific, inducing the transfer of knowledge, the interaction between what students know, the new information they encounter, and the activities they engage in as they learn.

The successful implementation of science curriculum requires effective planning by schools and teachers. Thus, teaching is a mechanism of delivering the lesson to the learners and a purposeful means to help students learn. Concept maps can be used in the teaching-learning processes [2]. In teaching science, the teachers require content knowledge, general pedagogical knowledge, curriculum knowledge, pedagogical content knowledge [3]. Seen at classroom level planning, teachers are required to prepare long-term and short–term plans that are based on the
curriculum, and incorporate progress records, planning for individual difference, balance between knowledge and skills, allocation of appropriate time for activities, role of the pupils, text book.

In modes of instruction different contents need different strategies, different learning styles mean students learn better with some methods than others, and a varied repertoire keeps students engaged and interested in the subject matter. Matching teaching strategy to learning need should be done. Continuous monitoring of this alignment between teaching strategies and learning assures that teaching evolves to match changing student learning needs. Hence, the priority of the teachers is to match the learning needs of students to appropriate teaching strategies in order to maximize quality and quantity of student achievement. The teachers have to be both knowledgeable in their content areas and extremely skillful in a wide range of teaching approaches to cater for the diverse learning needs of every student.

The concern of quality of education has direct relevance to universal and countrywide plan. Even though, it does not have a universally agreed definition, it is an elusive, relative, multidimensional and dynamic concept. Accordingly; quality teaching leads to higher achievement and close the gaps [4]. Quality as a concept comprises both descriptive and normative characteristics [5]. Therefore, quality may be an attribute of an individual or organization in addition to the status or relative degree of worth. Three approaches are identified to defining quality; the learner centered-input-process-output and multi-dimension, but UNICEF (2000) and UNESCO (2004) recognize five dimension of education quality namely, the learner, environment, context, priorities and outcomes as cited by [5]. UNESCO’s vision for global education depicts quality as learning to know, learning to do, learning to live together and learning to be. Likewise, EFA Global Monitoring Report described quality education in terms of the tangible input, process and contextual factors the affect quality of outputs cited by [5].
Thus, quality teaching is at the center of schooling systems and school effectiveness, and recognition of the critical relationship between teachers and learners highlights the need to better define and communicate what constitutes good teaching [6]. Moreover, Improvement in access and quality of primary education has been one of the central focus areas of development for Ethiopia in the past decades. Specifically, in Ethiopia since 2006; significant achievements have been made in expanding access to primary education service for local communities through decentralized service delivery mechanism. Accordingly, PBS and GEQIP are among the key project that support expansion and improvement in quality of education service at decentralized levels [7]. Amare (2011) examine the extent to which the Ethiopia primary education system provides quality education to young lives, in particular and the Ethiopia primary schools pupils, in general as cited by [5]. But, the finding showed that neither students nor teachers were sufficiently engage in active methods of learning [5]. Similarly, Harlen et al. (1995) have pointed towards problems linked to primary teacher’s lack of confidence in teaching science and their insufficient scientific knowledge background as cited by [8]. On other hand, Harlen (1996) commented that it might appear too difficult to find out the ideas of all the children in a class in such away as to plan activities to accommodate them as cited by [8].

A quality teacher is one who has a positive effect on student learning and development through a combination of content mastery, command of a broad set of pedagogic skills, and communications skills. Hence, Teacher‘ quality is one of the most important school factors in influencing student’s achievement (Darling-Hammond, 2000, 2003; Santiago, 2002) cited by [6]. Hence in Australia, quality of teaching and learning is an important factor accounting for variations in school students’ achievements (Cuttance, 2001: Rowe, Turner, and Lane, 2002) as cited by
Because, effective science teaching helps students develop conceptual understandings and inquiry abilities necessary to be productive citizens and science learners.

Inconsistently; many parents invest in their children`s education, expecting that quality education will bring significant transformation in their personal and social development as cited by [9]. These show that the teachers must be equipped with sufficient knowledge; skills and awareness in order to carry out their jobs and the determinants of quality. Thus, general education quality improvement program project was designed as a package to address quality in education comprehensive approach mainstream cross cutting issues.

In SIP Blue print [10] teaching and learning is the first domain set for students` result improvements. In such away mastery of subject content and methods, effective teachers` effort and commitment, and students` learning are the key elements in the domain. In this direction the study is in this bench mark of this element on the instructional process in the class room in primary school science subject practice.

From this blue print the mastery of subject content and methods, effective teachers` effort and commitment, were important for improving students results. Hence if these were in class room instructions it expected to promote MLC according to MOE students` profile. The following principles were expected from science teachers while conducting science instruction.

![Figure 1:1 Framework of classroom instructions](image-url)
Both constructivist and social theory of learning were concerned in the framework. In general the package’ programs were introduces and agreements across each stakeholders made and the implementations were carried out for more than a years. Additionally, in schools different curricular and co-curricular activities were done by science teachers. But stills there are parents’ questions on quality of instruction and, MOE struggle through schools’ organization, stake holders, and EBC media to avoid the cheating of students. Similarly, the teachers themselves negatively criticized the students` learning. But, the Country focused on science and mathematics education intensively and extensively for sustainable growth and to have well qualified human power. Contrarily, the interest of our trainees to join science stream were become struggle. Thus, study the science classroom instructions was found to be important and also the right time.

PURPOSE, RESEARCH QUESTION AND METHODOLOGY OF THE STUDY

The main purpose of this study was to investigate the status science classroom instruction in students` learning science in primary schools of Oromo Zone, ANRSEB, KCTE Linkage schools.

In this direction, this study was aimed at answering the following basic questions.

• How natural science classroom instruction succeeding the learning science of students` at classroom level?

• To what extent science classroom instruction fill the gaps of learners learning science?

The population of this study was lower grade primary schools science teachers. In such away, the targets of this study were grade three, four and six primary school sciences teachers who were teaching currently in the school now in 2016. The instruments to collect data were both developed adopted. Thus, Questionnaire, Interview, Observation followed by checklists and recording would
be employed to gather relevant data for this study. The data analysis methods were selected to fit both quantitative and qualitative data types. For qualitative data thematic analysis methods was selected, while percentage and mean and portrayed using figures i.e. statistical methods would be used to analyze the quantitative data.

PRESENTATION AND ANALYSIS OF DATA
Aspects of Planning of Science Teachers

![Bar chart showing aspects of planning of science teachers]

Fig. 1. Aspects of planning of science teachers

From the above the expected mean is 30 and the obtained mean is 24.2 with 8.3 for 4 and 15.9 for 3, and 8.8 with 5.3 for 2 and 0.5 for 1. i.e. of 30 respondents 81% them prepare instruction activities appropriately but 19% have gap planning instructional activities.
Science Teachers Lesson Delivering Reflection Response

![Chart](chart.png)

Fig. 2. Science teachers lesson delivering reflection

From the above figure 2 the expected mean is 30 the observed mean 20.4 with 6.2 for 4 and 14 for 3, and 9.6 with 8.8 for 2 and 0.8 for 1. i.e 68% science teachers response show that they deliver science instructions accordingly where as 32% of them claim their instructions delivering quality.

Student’s Science Learning

![Chart](chart.png)

Fig. 3. Student’s science learning
As can be observed in figure 3 above, the EM is 30 and the OM is 15.7 with 5.5 C and 10.2 O, and 14.3 with 11.5 S and 2.8 R; i.e. 63% science teachers’ response show that their students engaged in science learning where as 37% of them agree that there is a problem of students’ science learning (in both scientific knowledge and process) in their instructions. In such a way; on average 66% (20) science teachers argue that often their students engaged in science inquiry and 49.333% (14.8) of them argue that often their students engaged in scientific process.

**Insinuation of Teaching Aids in Realization of the Lesson**

![Insinuation of teaching aids used](image)

Fig. 4. Insinuation of teaching aids

From the above figure, the insinuation of teaching aids in realization of the instruction, the average expected mean 30 and the average observed mean 22 with 7.75 for 4 and 14.5 for 3, and 8 with 7.5 for 2 and 0.75 for 1. i.e. the teaching aids that the teachers used help the students’ active students’ involvement in instruction (23), in increasing content coverage (22), in addressing different
form of knowledge(22), and in promoting the high level of success of the instruction(21). Incase, 27% science teachers agree that their utilization of teaching aids didn’t realize their instruction.

**Subject Matter Knowledge**

![Subject matter knowledge](image)

Fig. 5. Subject matter knowledge

From figure 5, 67% teachers response show that they didn’t face content difficulty in instruction where as 33% face content difficulty in there instruction.

**Science Teacher’s Pedagogical Skill in Planning Science Activities**

![Pedagogically planning skills](image)

Fig. 6. Pedagogical skills in planning science activities
From figure 6, the science teachers have gap in planning participatory and SMART plan. The EM 30 and OM is 6. Hence; 27% (8) of science teachers have SMART lesson plan, 3% (1) plan for integration, No one plan for individual needs, 50% (15) of them differentiate activities in their lesson plan. Therefore, 80% science teachers have gap of pedagogical instructions planning.

Science Teachers Subject Matter Knowledge

![Subject Matter Knowledge Graph](image)

**Fig. 7. Subject matter knowledge**

From figure 7, the EM is 30 where as the OM are 1.3, 7.7, 18.3 and 2.7 respectively with 4, 3, 2, and 1. This show that 70% (21) teachers teaching science have gap of subject matter knowledge. So that, on average, 9% science teachers have subject knowledge gap in delivering science instruction.
Science Teachers Pedagogical Practice

From the Figure 8 on science teacher’s pedagogical practice, EM is 30 and the OM is 1.2, 6.2, 12.4, and 10.2 respectively with 4, 3, 2, and 1. These show that, on average 75 % (22.6) science teachers have gap in professional practice.
Types of Teaching Aids Used

From figure 9, 90% (27) science teacher use text book only for instruction, 7% (2) use picture and 1(3%) use real teaching aids for realization of the instructions. This show that, science teachers teaching aids utilization in realizing the instructions were low. The instructions were text book based only.

Methods of Teacher Teaching

Fig. 9. Teaching aids

Fig. 10. Methods of teacher training
Figure 10 shows that group discussion and lecture methods were used by most science teachers; i.e. 30 and 29 respectively whereas answer-question and practical teaching were used by few teachers i.e. 5 and 1 respectively. So that, most science teachers’ teaching methods were weak in learning science by doing.

Schools Principals` Response on Science Classroom Instruction

![Graph showing data]

Fig. 10. Principals’ responses

Figure 11 shows that schools principals’ response EM is 8 but the OM is 2.8, 2.8 and 2.4 for 0.95, 0.75, and 0.5 respectively. According to schools` principals, on average 30% science teachers have gap in delivering science instruction properly.

The analysis data obtained from instruments used reveal that a number of primary school science teachers have gaps both in mastery of subject matter and methods, and in exploiting effort in succeeding students` science learning. In such way, the challenges were pedagogical knowledge, exploiting the effort to professional practice and subject matter knowledge. It is thus learnt that science class room instructions in lower grades of primary schools were not in position of
developing scientific skills and processes of the children. This assertion can also be deduced from the following table that contains the expected and observed mean values.

Table 1: Summary of Expected and Observed Mean Values

<table>
<thead>
<tr>
<th>Teaching activities</th>
<th>Teachers response results</th>
<th>Schools response results</th>
<th>Observation results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>EM</td>
<td>OM</td>
<td>EM</td>
</tr>
<tr>
<td>Mastery of Subject content</td>
<td>30</td>
<td>20</td>
<td>8</td>
</tr>
<tr>
<td>Professional practice</td>
<td>30</td>
<td>20.575</td>
<td>8</td>
</tr>
<tr>
<td>Exploiting effort to under rooting instruction by integrating subject knowledge with professional quality</td>
<td>8</td>
<td>5</td>
<td>8</td>
</tr>
</tbody>
</table>

CONCLUSIONS AND RECOMMENDATIONS

The purpose this study was exploring the quality of natural science education classroom instruction in lower grades of KCTE linked primary schools. The framework for the quality instruction was SIP which has been implemented in the schools for the last six years. The target of the population is primary school science teachers who were teaching in lower grades of primary schools. Of 14 associated schools, eight of them were selected based on accesses to transportation and proximity. The grades were selected using probability sampling using lottery methods. Sections and the school principals were selected using availability sampling methods.

The instruments, the questionnaire and observation checklist, were both developed and adopted from the effective instructions and the elements of teaching – learning domains of SIP. The procedures of data collection were administering the questionnaires, interview and observation supported by checklist and video recording.
The variables were mastery of subject matter and methods, and exploiting effort for integration of subject knowledge and pedagogical practice for delivering instructions.

The results obtained from the questionnaires, observation checklist and recording, and interview show that science classroom instructions have a problem. It was also observed that science teachers have gaps in subject matter knowledge, pedagogical planning and practice, in exploiting the effort for integration of them in delivering.

From this study we conclude that the primary schools lower grade science teachers classroom instructions were not in a position of succeeding the learning science of students and filling the gaps of learners in science learning.

Based on the findings, the following recommendations are forwarded.

- KCTE department of natural science educators should give workshops based on selective topics of science contents for the primary schools science teachers, science practical teaching, pedagogical planning, the way of delivering science instructions.
- Schools science department heads, principals and cluster supervisors should work together to support and make close monitoring of classroom instructions and awakening of science teachers teaching.
- Science teachers should make professional dialogue among themselves through the departments on the way of science delivering and the contents delivered.

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CHEMISTRY AT ELEMENTARY SCHOOLS: PARTICLES OR ALSO ATOMS – THAT’S THE QUESTION

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ABSTRACT
At elementary school level, the description of substances and their changes is preferred by most guidelines of education. If the elementary school includes fifth and sixth graders like in Africa, it seems to be possible to introduce the idea of the Particle Model of Matter to reflect changes of states, dissolving and diffusing processes. As an example the solution of sugar in water can be represented by a mixture of spheres and crosses as in the figure below [1]: one sphere shows a sugar particle, one cross shows one water particle. The only difficulty is the scientific language concerning the original sugar solution, the mental model in the cognitive structure, and the kind of concrete models – may be spheres and crosses or circles with different colors. [African Journal of Chemical Education—AJCE 7(2), July 2017]
INTRODUCTION

The introduction of the Particle Model of Matter matches with the famous Chemical Triangle of Johnstone [2]. He points out that we have to differentiate the macro level of substances and their chemical reactions from the interpretation on the sub-micro level by simple particles or by atoms/ions/molecules, and the description by formulae and chemical equations on the representational level (see figure 2). The only way to understand Chemistry is the sub-micro level: without interpretation on the second level, the formulae and equations on the third level must be memorized without understanding. For elementary schools, the first and second level can be realized – the third level concerning formulae and equations should be moved to instruction in junior and senior high schools.

Fig. 1: Terminology on the level of substances, mental models and concrete models [1]
The explanation of solution and diffusion processes can be done with the particle model of matter – the explanation of chemical reactions, specially the combustion process, cannot be interpreted with particles. The big question is now if atoms and molecules can be introduced successfully in elementary schools for 5th and 6th graders to reflect the important topic about combustion reactions. One example: if the combustion of carbon will be discussed, you need Daltons atomic model to talk about C atoms, O₂ molecules and CO₂ molecules (see figure 3). It
should be teacher’s decision if he or she knows the pupils so well that he or she can work successfully with Dalton’s model of matter: particles or also atoms – that is the question!

**CHEMISTRY FOR LOW GRADES 1 – 3**

For pupils of grades 1 – 3, the teacher should show only substances and phenomena of nature, the kitchen and the laboratory: properties of pure substances and their changes by heating with the burner. Dissolving processes may be demonstrated or done by the pupils themselves. The investigation of mixtures and the separation of mixtures to pure substances are easily done and described by every-day language. Some examples:

**Phenomena in nature:** water evaporation and water steam condensation with discussion of weather phenomena, observing burning processes of wood, coal and fuel – looking to the role of air, discussing sun with heat and light energy, interpretation of photosynthesis processes on a reduced level, looking to pieces of noble metals, ores, minerals, etc.

**Phenomena in the kitchen:** investigating white powders like sugar, salt, starch, flour and baking powder; boiling and freezing water by observing the thermometer; weighing special portions of flour and sugar, baking and cooking, investigating liquids like milk, oil and vinegar, discussing electrical energy and the transfer into heat by a water-heater or by other cooking facilities; transfer into heat and light by the bulb in a lamp, etc.

**Phenomena in the laboratory:** melting and boiling temperatures of some pure substances; density of some metals, electric conductivity of metals and salt solutions, dissolving salts until the saturated solution occurs, dissolving gases in water, looking to the mass of 1 L of air (1 L of air weighs 1.3 g); showing the bigger volume of heated gases compared to normal temperature ("coin
on the bottle neck, hands on the bottle”), investigating mixtures and separating pure substances, observing energy transfers from the burner to water, etc.

If possible, the young kids should do many experiments by themselves. In that age kids are very curious and really motivated to do something: instead of “chalk and talk”, famous experiential learning and learner-centered education should be realized.

If teachers want, the particle model of matter can be introduced in a very simple way (states of matter, solutions) – but normally the concrete models have only a small chance to be understood by those young kids. The proposal of models will follow for grades 4 – 6.

CHEMISTRY FOR HIGH GRADES 4 – 6

The 4 – 6 grades seem to be ready for understanding the mental model that matter is composed of small particles which we can never see: water by water particles, sugar by sugar particles. They also should be ready for taking spheres, coins or crosses as models to represent those particles by a concrete model (see figure 1). They may also be ready for working with Dalton’s atomic model: carbon contains of C atoms, oxygen contains of O atoms combined to O₂ molecules, which may react to CO₂ molecules (see figure 3).

1. Particle Model of Matter

The explanation of chemical changes should start with the particle model of matter. Experiments are shown on the macro level and should be interpreted by particles or groups of particles on the sub-micro level (figure 2).

One example by the famous experiment “bottle with jumping coin” [3]. In grades 1 – 3, the pupils have taken an empty bottle (full of air!), given some drops of soap solution on the
opening and a coin on it. When pressing the warm hands on the outside of the bottle, the coin starts “to jump up and down” several times. Pupils will explain this observation by the idea that “warm air takes a bigger volume than cold air, the volume increases by heating the air”. The 5 – 6 graders will interpret this explanation by the particle model of matter: air particles are moving all the time and occupy a special volume, after heating the bottle air particles are moving faster and the distances increase from particle to particle – the volume becomes bigger than before. Other experiments and model interpretations follow, pupils should discuss and draw their mental model:

a) **Heating mixtures of ice and water**: watching the thermometer which stays constantly by 0 °C, ice shows water particles in a regular and symmetric order, energy is used for separating the particles in water which have after heating no order and move fast all the time.

b) **Boiling water, measuring the temperature of water and water steam**: both temperatures show the same value of 100 °C. In water the particles are moving all the time but stay together, by heating the distances between the particles grow, the particles are moving faster – water steam takes a much bigger volume than the same portion of liquid water.

c) **Dissolving oil and ethanol in water**: oil is not soluble in water (but in gasoline), ethanol is very good soluble in every ratio with water, water particles and ethanol particles are moving and mixing together. Distilling red wine: a colorless liquid mixture of ethanol and water comes out, water particles and ethanol particles are separating from pigment particles and other particles of red wine.

d) **Diffusion of two gases**: a cylinder is filled with brown bromine vapor and covered on the opening by another colorless air-filled cylinder. After some minutes both gases will mix and make a light brown gas mixture, bromine particles and air particles are moving all the time, are colliding and mixing.
e) Making “gold from copper”: a mixture of zinc powder and hot concentrated sodium hydroxide solution is prepared (caution – goggles!). A coin with a copper surface is dipped into the solution: the copper turns silvery, “silver is produced”. After drying the coin and heating it in a colorless flame, the color of the coin turns golden, “gold is produced”. Zinc particles are settling on the coin surface (silvery color), in the heat of the flame zinc particles and copper particles are mixing to a well-known alloy: brass with golden color.

Other experiments may follow which can be interpreted by the particle model of matter. But this model fails if chemical reactions are involved – Daltons atomic model may be introduced and will be able to interpret simple reactions – specially combustion processes.

2. Daltons atomic model of matter

John Daltons big theory of the year 1808 was the connection of well-known elements with the idea of atoms: carbon should be composed of C atoms, copper of Cu atoms, etc. The atoms of one element should have the same mass, different elements are composed of atoms with different masses: m (H atom) = 1, m (C atom) = 12, m (O atom) = 16. The first table of atomic masses came out and Chemistry moved from alchemy to the real science.

The big advantage of Dalton’s model is to explain the law of conservation of mass: no atom can be destroyed, no atom can be created, the involved atoms only regroup by reactions (see figure 3): C atoms in the graphite lattice and O₂ molecules of oxygen regroup to CO₂ molecules in carbon dioxide. If the teacher decides to introduce Dalton’s model of matter, he/she may show some important atomic symbols (H, C, O, Fe, Cu, S) and some molecular symbols (O₂, H₂O, CO₂) with the help of spheres of different colors and sphere groups for molecules (see molecular model set)
or dense packing of spheres for metal crystal lattices. Students will interpret first chemical reactions with the help of their teachers. Examples:

a) **Burning pieces of charcoal in a closed flask**: 4 – 6 little pieces of charcoal are taken in a 1-L-round flask, which is filled with oxygen. The flask should be closed by an air balloon and weighed. The pieces are heated with a burner from outside. After glowing the flask should be moved around and the coal is burning brightly. After cooling down, the closed flask is weighed again: same mass as before. The gas in the flask is tested by limewater: carbon dioxide. This gas portion weighs the same as carbon and oxygen before: C atoms and O\(_2\) molecules are regrouping to CO\(_2\) molecules (see figure 3).

b) **Burning iron wool in an open and closed system**. Two big bunches of iron wool (same mass) are hanging at a balance, one bunch should be ignited by a burner flame: this bunch goes down, turns from grey to black color, the mass increases. Another bunch of iron wool is taken in a test tube, which is closed with an air balloon and weighed. With the strong burner, the test tube is heated until the iron wool glows and turns to black color. After cooling down, the test tube is weighed again: same mass. Reacting Fe atoms and O\(_2\) molecules to a crystal lattice of Fe and O atoms can be interpreted with a model drawing (see figure 4). In a closed system, the law of conservation of mass can be shown. The first experiment with an open system shows the increase of mass by reacting oxygen of the air to form black iron oxide.
c) Reacting copper plates in sulfur vapor. In a test tube, sulfur is heated until sulfur melts and the tube is half filled with brown sulfur vapor. The copper plate (about 1 x 5 cm) is heated in the colorless flame and put down into the brown liquid and vapor: the copper glows red and change to a black substance, called copper sulfide. The interpretation can go from Cu atoms in a crystal lattice and from S atoms in the molten sulfur to a regular lattice of Cu atoms and S atoms in copper sulfide (see figure 5).

**MISCONCEPTIONS OF YOUNG STUDENTS**

For some science topics, students are bringing alternative conceptions or pre-concepts or misconceptions which are developed from phenomena in every-day life or by language problems. One example. If all people are mentioning after a big rain and the appearing sun that “water puddles
on the street disappear, the sun have taken the water away”, the young students develop a distorted concept: “substances can disappear without any rest, they can be destroyed irretrievably“. No kid will develop the idea that water evaporates and water steam is mixing with the air, that water particles are moving, escaping from the water surface and are mixing with moving particles of the air.

So science teachers have to challenge those pre-concepts with experiments and concrete models of involved substances. They should give the students the chance for a conceptual change from their mental model to the scientific model. Because the student has developed his or her mental model over years, a conceptual change is not easy: the teacher has to take more experiments and concrete models to change the mental model in the brain of the student – one hour of lecture cannot change the pre-concept, the change needs weeks and months. Otherwise, by the language of people (“after burning charcoal on the grill the charcoal is away”), the pre-concept may occur again.

Sometimes concept cartoons help (see figure 6): concerning the composition of water steam three persons are thinking in non-scientific ways like “water changes to air, to hydrogen gas and oxygen gas”, only one person gives the right answer: “it changes to water steam” [5]. For many topics, some misconceptions are observed, and their challenge is offered [6]:

a) “Gases weigh nothing, air is surrounding us, hot air is even going up”. Teachers have to show by experiment that 1 liter of air has the mass of 1.3 g, that the air of the classroom can weigh thousands of kilograms, that the atmosphere with the altitude of more than 20 km of air makes a special pressure on us which can be measured by a barometer.
b) “Carbon is after combustion away, is irretrievably destroyed”. Students cannot observe the gas oxygen, and cannot see the produced colorless gas carbon dioxide after the burning process. Teachers have to show the reaction of carbon with oxygen, have to prove the formed gas with limewater and may visualize the law of conservation of mass, pointing out that C atoms and O$_2$ molecules are regrouping to CO$_2$ molecules of the same mass (see figure 3).

c) “Burning iron wool changes the color from grey to black”. Teachers have to realize the experiment with iron wool on a balance to show that oxygen is reacting and forming a new
substance – iron oxide. This new substance, iron oxide, has the black color; iron metal is all the time grey and cannot show another color.

d) “Energy is gone”. People are discussing their electricity bill and speaking of "used energy which is gone". The students have no chance to discuss that energy cannot be destroyed, that energy can only be transferred from one form to another form: i.e. electrical energy at home can be transformed to light energy and heat energy in the bulb of a lamp.

To challenge the misconceptions, teachers have to do experiments instead of “chalk and talk”: experiments convince students, and together with models of matter, the conceptual change can be realized. But it takes time and more experiments and models of the same content – otherwise students stay with their misconceptions and will not discuss scientifically in their future. Teachers have also to pay attention to the language by working with models (see figure 1).

There is especially in Chemistry a “laboratory jargon”, which is not helpful for scientific discussions: “water contains of two hydrogens and one oxygen” is sometimes the comment of a chemist. He/she hopes that his/her colleague will understand the “slang” which means correctly: “one molecule of water is composed of two H atoms and one O atom”. Experts may stay with the laboratory jargon – for Chemistry education we have to take the scientific terminology: the students are learners and cannot interpret any slang [7].

**METHODS OF INSTRUCTION**

Presented contents in class are not easy to understand. If the teacher is only presenting the topics, filling the blackboard and asking the students to copy it into their folder, students have a hard work to understand. Students should work themselves with contents and should present their own ideas to explain phenomena. So the teacher has to prepare worksheets or group work to
include students in the learning process; he/she should realize learner-centered education. If possible, students may perform their own experiments, may develop their own mental model and build or draw their own concrete model for special phenomena.

1. Cognitive conflict

Students should be motivated to think about chemical phenomena – one way is the cognitive conflict. When the teacher burns a piece of iron wool and asks about the mass before and after burning, most students will answer from experiences in every-day life: “after burning the iron wool weighs less than before”. If the teacher takes now two bunches of iron wool at the balance (see 2.c) and shows that the burning bunch gets heavier, students will have a cognitive conflict: “why heavier?”. After long discussions, the reason should be the surrounding air, oxygen as a part of air is reacting to produce black iron oxide, iron oxide weighs more than the bunch of pure iron wool before.

2. “Think, pair, share”

For many problems, it is advantageous when teachers give a problem into class and ask students to work first alone and think about an explanation, then to work together as a pair with the neighbor in class, finally some of those pairs present the explanation to share it for the whole class until the right solution is accepted by most of students. After demonstrating the experiment “iron wool at the balance”, students may think alone about the observation “heavier”, then he or she goes together with his or her neighbor to solve the problem as a pair, finally the whole class is sharing the results of the pair work.
3. **Group work**

By working with experiments to show the melting process of different substances, groups of students may investigate different substances. Every group gets a burner, a test tube, a thermometer and a teaspoon of a substance: sugar, salt, sulfur, naphthalene, stearin, etc. The groups try to find the melting temperature and can also look to the thermometer during the melt gets solid: the temperature stays for the time constant when both solid and liquid substances fill a part of the test tube, after all substance is solid the temperature goes down to room temperature. Every group can present their results for the whole class.

4. **Learning stations**

This is another type of group work. When there is a topic with different aspects, different experimental stations can be planned. If salts may be investigated, the teacher can install following stations on different tables in the classroom: melting kitchen salt and sodium nitrate in a test tube (first salt melts at 800 °C, the other one at about 310 °C), solubility of salt (how many grams of salt are soluble in 10 mL of water?), separation of salt from ocean water (4 % solution of salt in water should be prepared), density of saturated salt solution (what swims on water and on saturated salt solution?), separation of a mixture of salt and pepper or sand, etc. Student groups have to switch from station to station to learn much about salts, they are motivated by doing experiments themselves, by moving around the classroom and discussing the observations with friends. The role of the teacher is the advisor.

5. **Research**

Because it is not easy to instruct young kids in science, we have to evaluate our steps of education. One research question may be to look which is the most successful way of instruction:
without experiments, with demonstrated experiments by the teacher, with hands-on experiments by students themselves.

When the particle model of matter, is introduced we can compare classes that work with this model and without the model, which work only by talking about particles and those which build or draw models of the observed phenomena. When Dalton’s atomic model will be introduced in grades 5 or 6, it is a must to research how they are able to grab the idea of atoms and molecules by special reactions. Because much people are refusing this early introduction, we should have the results of research to convince those people by our facts.

Also the methods can be observed scientifically. Because many teachers like to “chalk and talk” without including discussions with students, we should prove that methods like “think, pair, share”, group work and other learner-centered lessons are bringing us more success and also more motivation of students compared to teacher-centered education.

Even motivation can be evaluated by empirical research. If we ask what students like in science or chemistry education, we may find experiments and specially students experiments are very motivating, also investigating things out of the kitchen and out of every-day life motivate students more than working with unknown substances of the laboratory. All these questions can be evaluated by research and empirical investigation.

CONCLUSION

Not many curricula of elementary education propose Dalton’s atomic model of matter to explain chemical reactions. If African schools will do it for grades 5 – 6 and will evaluate this way of instruction by empirical research, it will be internationally a big step for modern Chemistry education. But success can only be proved if students work with concrete models like drawings or
spatial models of chemical structures for involved substances: students will develop their mental model from concrete models – they will never realize it by “chalk and talk” or only by talking about particles without those concrete models.

All proposed instruction steps are value for student groups in junior or senior high schools: teachers may take the particle model of matter and later Dalton’s atomic model. If they do not want to switch from one model to the other, they are also free to skip the particles and come just to atoms and molecules by the atomic model. It is even possible to introduce the existence of ions by an extended atomic model to describe salts, acids and bases correctly by involved ions – we have not to wait for the nucleus-shell model of atoms [8]. When we realize instruction on the submicro level, students will understand Chemistry and will be motivated to go on with this interesting science.

REFERENCES
ASSESSMENT OF THE AVAILABILITY AND USABILITY OF INFORMATION AND COMMUNICATION TECHNOLOGY (ICT) RESOURCES IN CLASSROOM DELIVERY BY SECONDARY SCHOOL CHEMISTRY TEACHERS

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ABSTRACT
This research is to find out whether ICT facilities and resources for classroom delivery were available in secondary schools in Benue State, and whether Chemistry teachers in those schools were utilizing the resources in their classroom delivery. The design is a survey. Thirty six (36) Chemistry teachers randomly sampled from all the secondary schools in Benue State formed the sample. A structured questionnaire was used to collect data for the study. Percentages, means and standard deviations were applied to analyze the data. Analysis of the data revealed that almost all the ICT facilities and resources for teaching Chemistry were not available in the schools used for the study. In addition, Chemistry teachers in those schools were not using ICT resources in their classroom delivery. It was concluded that ICT resources were not being used in teaching Chemistry in secondary Schools in Benue State. The researcher therefore recommended, among others, that effort should be made by the governments, non–governmental organizations, parents–teachers association and philanthropists to provide ICT facilities and resources in all the secondary Schools in Benue State. [African Journal of Chemical Education—AJCE 7(2), July 2017]
INTRODUCTION

A recent development in science education and virtually other fields is the application of information and communication technology (ICT) [1]. ICT is said to be a range of technology for gathering, storing, retrieving, processing, analyzing and transmitting information. It encompasses a wide range of technology like Telephone, Computer, Satellite, Telex, Fax, Radio, Television, Software, Hardware, Projectors, Video and Bulletin board to mention but a few [2]. The application of ICT in classroom delivery has been seen to have positive impact in the achievement of students since it has the capacity of increasing and sustaining the interest of students in the teaching and learning of every subject, especially science subjects [1].

Chemistry being one of the pure science subjects is termed a central science [3]. Chemistry permeates into other science subjects, both pure and applied, to give them value. In everyday living and in academic pursuit, Chemistry plays dominant roles. Audu [4] made it clear that Chemistry is seen in everything we do daily in our homes such as cooking, washing, cleaning and so on. In academic pursuits, reading any science course in the tertiary institutions require a credit pass in Chemistry [5]. This makes Chemistry to be the queen of all the sciences. It is not an over statement to say that Chemistry is science and any other science subject is an applied Chemistry.

Nevertheless, there has been a consistent downstream achievement in Chemistry among secondary school students in different states in Nigeria including Benue State [6-8]. Omituyaki [9] noted that the percentage of students that pass Chemistry at credit level in senior School Certificate Examination (SSCE) has never reached 40% in the recent past. Note that this percentage, less than 40, is the percentage of candidates who qualify for higher degree admission in science related courses, other things being equal.
The long-standing consistent poor achievement of secondary school students in Chemistry in Nigerian secondary schools, especially in secondary schools in Benue State is a threat to Nigerian economic, social, scientific and technological development. This ugly trend has been a worry to concerned Nigerians (educationists, scientists, policy makers and the like) [10]. Recent research reports show that the major factors leading to this poor achievement include lack of interest in the teaching and learning of Chemistry among the students, which emanates mainly from the monotonous and ineffective methods of instructions used by the teachers, lack of skilled professional teachers, intellectual demand of the subject, lack of instructional materials among others. Standing out among these factors is the issue of ineffective method of instruction used by teachers.

British Educational Research Association (BERA) [11] acknowledged widely that the most important factor influencing students’ achievement is high quality teaching. High quality teaching is a product of skilled professional teachers among others [12]. It is the teacher who translates the curriculum into practice. Thus it was stated that teacher education should provide the teachers with the intellectual and professional background adequate to any changing situation, not only in the life of their country, but in the wider world [13].

ICT application in classroom delivery is a new approach for teaching and learning globally in which it is necessary to create more access to information and experience through global networks and pool of knowledge [1]. In the classroom usage ICT provides both learners and teachers up-to-date information in their field of study, brings to their doorsteps latest scientific discoveries and research reports from all over the world [2]. Since the use of ICT in classroom-delivery has the potential of increasing and sustaining the students’ interest in the teaching – learning process, one can say that using ICT in teaching chemistry in secondary schools in Benue
State can improve the achievement of the students in the subject. However, Asongo [2] made it clear that for the teacher to effectively use ICT in teaching learning, the resources and facilities for ICT should be available in schools and the teachers should be proficient in using them often in classroom delivery. It becomes necessary therefore to find out whether secondary schools in Benue State have ICT resources and facilities and whether the Chemistry teachers in those secondary schools utilize these resources often in classroom delivery.

**THE RESEARCH QUESTIONS, METHODS OF DATA COLLECTION AND ANALYSIS**

This study seeks to find answers to the following research questions:

1. What are the ICT resources and facilities available for teaching chemistry in secondary schools in Benue State?
2. How often do chemistry teachers in secondary school in Benue State utilize the available ICT resources in their classroom delivery?

The research employed a survey of the availability of the ICT resource in the secondary schools in Benue State and how often chemistry teachers make use of these resources. All the chemistry teachers in secondary schools in Benue State make up the population for the study. From this population, 36 chemistry teachers were randomly selected to serve as samples for this study.

A structured questionnaire was used to collect data for the study. The data was analyzed using means, standard deviations and percentages.
FINDINGS AND DISCUSSIONS

Research question 1: What are the ICT resources and facilities available for teaching chemistry in secondary schools in Benue State?

Table 1: ICT Resources and Facilities Available for Teaching Chemistry in the secondary schools

<table>
<thead>
<tr>
<th>S/n</th>
<th>Resources and facilities</th>
<th>Percentage(%) availabilities</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Computer</td>
<td>25</td>
<td>Not available</td>
</tr>
<tr>
<td>2</td>
<td>Printers</td>
<td>75</td>
<td>Available</td>
</tr>
<tr>
<td>3</td>
<td>Television</td>
<td>10</td>
<td>Not available</td>
</tr>
<tr>
<td>4</td>
<td>Video Cassette</td>
<td>0</td>
<td>Not available</td>
</tr>
<tr>
<td>5</td>
<td>Scanner</td>
<td>28</td>
<td>Not available</td>
</tr>
<tr>
<td>6</td>
<td>Storage disk</td>
<td>35</td>
<td>Not available</td>
</tr>
<tr>
<td>7</td>
<td>Internet facilities</td>
<td>15</td>
<td>Not available</td>
</tr>
<tr>
<td>8</td>
<td>Projector</td>
<td>0</td>
<td>Not available</td>
</tr>
<tr>
<td>9</td>
<td>Good Hand Sets</td>
<td>25</td>
<td>Not available</td>
</tr>
<tr>
<td>10</td>
<td>Radio</td>
<td>10</td>
<td>Not available</td>
</tr>
<tr>
<td>11</td>
<td>Steady electric light</td>
<td>5</td>
<td>Not available</td>
</tr>
</tbody>
</table>

Data in table 1 shows that all the ICT resources and facilities for teaching chemistry, except printers, were not available in the secondary schools used for this study. Looking at the table, only printer scores 75% in percentage availability showing that it was present in the schools used for the study. Other resources and facilities scored below 50% in percentage availability showing that they were not available in the schools used for the study. Asongo [2] found out that resources for internet accessibility were not available in most secondary schools in Nigeria. That this ugly situation has not changed is a thing of great concern. This is really a pathetic and worrisome situation. If ICT resources and facilities are not available in almost all Nigerian secondary schools, one wonders how the aims and objectives of teaching chemistry in secondary schools, which include:

- To equip students to live effectively in our modern age of science and technology.
- To enable students to develop reasonable level of competence in ICT application that will engender entrepreneurial skills, among others could be achieved.
Research Question 2: How often do chemistry teachers in Secondary schools in Benue State utilize ICT resources in their classroom delivery?

TABLE 2: The Usability of ICT Resources and Facilities in Classroom Delivery

<table>
<thead>
<tr>
<th>S/N</th>
<th>Resources/facilities</th>
<th>Mean</th>
<th>SD</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Computer</td>
<td>2.75</td>
<td>0.02</td>
<td>Often used</td>
</tr>
<tr>
<td>2</td>
<td>Printers</td>
<td>2.75</td>
<td>1.19</td>
<td>Often used</td>
</tr>
<tr>
<td>3</td>
<td>Television</td>
<td>1.00</td>
<td>0.00</td>
<td>Not often used</td>
</tr>
<tr>
<td>4</td>
<td>Video Cassette</td>
<td>1.00</td>
<td>0.00</td>
<td>Not often used</td>
</tr>
<tr>
<td>5</td>
<td>Scanner</td>
<td>1.35</td>
<td>0.65</td>
<td>Not often used</td>
</tr>
<tr>
<td>6</td>
<td>Storage disk</td>
<td>1.62</td>
<td>1.01</td>
<td>Not often used</td>
</tr>
<tr>
<td>7</td>
<td>Internet facilities</td>
<td>1.22</td>
<td>0.56</td>
<td>Not often used</td>
</tr>
<tr>
<td>8</td>
<td>Projector</td>
<td>1.00</td>
<td>0.00</td>
<td>Not often used</td>
</tr>
<tr>
<td>9</td>
<td>Good Hand Sets</td>
<td>1.23</td>
<td>0.12</td>
<td>Not often used</td>
</tr>
<tr>
<td>10</td>
<td>Radio</td>
<td>1.87</td>
<td>0.78</td>
<td>Not often used</td>
</tr>
<tr>
<td>11</td>
<td>Steady electric light</td>
<td>0.85</td>
<td>0.14</td>
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</tr>
<tr>
<td>12</td>
<td>Modem</td>
<td>1.00</td>
<td>0.00</td>
<td>Not often used</td>
</tr>
</tbody>
</table>

An examination of data in table 2 shows that chemistry teachers in secondary schools in Benue State often used only computers and printers. This is so because their mean values are above 2.50, which is the criterion mean. All other ICT resources and facilities used for the study were not often used by the chemistry teachers as can be seen from their mean values that are below the criterion mean. This finding is similar to earlier findings [1, 2] who noted that the teachers did not use most of the ICT resources for classroom delivery. It is not, however, surprising that teachers were not using the ICT resources. Table 1 shows that these resources were not available in the schools by the criterion of 50% availability in this study. Therefore, teachers cannot use what is not available to teach. It is instructive to note that teachers not using the ICT resources in their classroom delivery may not be as a result of lack of interest. Ikemelu [1] noted that using ICT in teaching and learning increases and sustains the interest of teachers and learners.

This finding has implication in the teaching and learning of chemistry in secondary schools. The objective of teaching chemistry in secondary schools, which aims at enabling students to
develop interest in chemistry and develop reasonable level of competence in ICT applications, may not be achieved.

CONCLUSION AND RECOMMENDATIONS

From the findings of the study, it can be concluded that most of the ICT resources and facilities for chemistry teaching and learning were not available in the secondary schools in Benue State. The study also reveals that chemistry teachers in secondary school in Benue State were not using ICT resources in their classroom delivery. Based on these conclusions, the following recommendations are made:

1. Effort should be made to provide ICT facilities and resources in all our secondary schools. This will be a joint effort of the governments, non-governmental organizations, parent teachers association, Philanthropist and so on.

2. Periodic training and re-training of chemistry teachers and other teachers on the usage of ICT facilities for classroom delivery should be embarked upon and sustained. These trainings need to emphasize the importance of ICT in the teaching and learning of every subject, especially science subjects. In addition, skills for effective usage of ICT resources in classroom delivery should be inculcated in the teachers.

3. Welfare packages for teachers such as science allowance, housing loan, medical allowance, prompt and regular payment of salaries and allowances should be made available for science teachers. This will make them to be happy on their profession and to devote time to acquire skills that will enable them to be effective on the job.
REFERENCES


SPECTROPHOTOMETRY MOLE RATIO AND CONTINUOUS VARIATION EXPERIMENTS WITH DITHIZONE

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ABSTRACT

The well-known color development dithizone reagent, H₂Dz, has been used extensively in trace metal analyses for almost a century. However, very little effort has gone into developing derivatives for aqueous media applications in absence of organic solvents. Synthesis of a suitable (p-COOH)H₂Dz derivative is reported here. For spectrophotometric determinations of various metal (M) to ligand (L) ratio’s in dithizonato metal complexes this reagent provides a versatile alternative for undergraduate chemistry practicals that is cost-effective, yielding repeatable results. The mole-ratio method yield a ratio of 1M : 1L for the silver dithizonate complex and 1M : 3L for cobalt. Employing the continuous variation method give M : L ratio’s of 1 : 3 for both nickel and cobalt. Formation constants are readily calculated from absorbance data. Complete methods, data, calculations and outcomes are reported here. All ratio’s are confirmed by mass spectroscopy.

[AFERICAN JOURNAL OF CHEMICAL EDUCATION—AJCE 7(2), July 2017]

Graphical Abstract
INTRODUCTION

Metal to ligand ratios and constants of formation are important aspects to consider during selection of suitable complexing reagents. Undergraduate analytical chemistry textbooks like Fundamentals of Analytical Chemistry describe three methods traditionally used for determining metal to ligand ratios in complexes [1]:

1. the continuous variation method,
2. the mole ratio method and
3. the slope-ratio method.

Without having to isolate complexes as pure compounds, both its composition and formation constants, $K_f$, may be determined by means of spectrophotometrical methods. This is possible since measurements are recorded quantitatively without disturbing reaction equilibria. Species that do not absorb are also readily determined, typically by using color development reagents like thiocyanate, 1,10-phenanthroline, or as reported in the present study, by reaction with the intensely colored blue-green carboxydithizone, henceforth referred to as the ligand, L.

Yoe and Jones introduced the mole ratio method, which is ideally suited for determination of complex stoichiometry [2-3]. Absorbances are measured for a series of solutions where in the metal concentration is kept constant while that of the ligand is systematically increased. Resulting plots of absorbance versus mole-ratio of reactants are characterized by two straight lines of different slopes that intersect at the equivalence point mole ratio, see Figures 1 and 2. Not just one, but also the formation of two or more complexes may simultaneously be identified, i.e. where successive slope changes occur, complexes have different colors or molar absorptivities, and constants of formation differ amply.
Formation constants may be obtained from absorbance data at the curved section of the plot. An increase in curvature is indicative of weaker complexes and therefore smaller constants of formation, which inevitably also increases uncertainty during identification of mole ratios [4]. Limiting factors are the stability of the formed complex, effect of the buffer and presence of masking reagents [5]. Techniques that may enhance application of this method to weak complexes are discussed by Chriswell et al [6].

It was however Job who originally introduced the method for determining formulas and formation constants of complexes [7-8]. The method was later modified, becoming the most widely used routine for determining composition of soluble metal complexes. Job’s method later commonly became known as the continuous variation method. A series of solutions are prepared where in the concentration of one reactant is systematically increased while the other is decreased; the combined concentration staying constant throughout. Solution absorbances are subsequently recorded at an appropriate wavelength. Provided that only a single complex forms, the plot of absorbance values against the volume fraction of one reactant is typically triangular in shape. Curvature at the apex is indicative of a complexation reaction that does not proceed to completion, see Figures 3 and 4.

In practical terms, the corresponding $K_f$ may be determined from the difference in absorbance between the said apex, and the intersect of two theoretical straight lines on both sides of the curvature that are extrapolated along the linear parts of the plot. Certainty in results is obtained only when one predominant complex forms, where the reactants and complex are not involved in associated equilibria [9].

A typical example where Job’s method is often applied is the iron-thiocyanate system. It however yields a variety of coordination ratio’s, depending on sometimes minute changes in
reaction conditions, like pH [10]. We found that results are consequently inconsistent amongst large groups of relatively inexperienced students, which poses a major challenge during undergraduate practicals. We therefore developed a new system utilizing a dithizone derivative that may complex several metals while requiring little additional reactants or costly organic solvents, see Scheme 1. Hereby the focus remains on the spectrophotometric method and not on synthesis. The carboxy derivative of dithizone unfortunately is not yet available commercially, but may readily be synthesized in large quantities and in high yield within less than a day. Due to the very intense color of dithizone only minute quantities are required during experiments employing both the mole ratio and continuous variation methods. For a student to execute and complete any one of the four experimental procedures reported here below, together with its set of calculations, is not expected to take more than 3 hours.

Scheme 1. Aqueous medium metal complexation with para-carboxydithizone.

Ever since the early 1900’s when Hellmut Fischer [11] introduced dithizone as new organic reagent in the field of analytical chemistry it has had a great impact on analytical procedures. These include various titrimetry applications, as well as mono- and mixed-color procedures applicable to trace metal analyses. Special features are its great sensitivity and considerable selectivity, the intensely colored complexes it forms with metals, its solubility in a range of organic solvents, and insolubility in water while readily soluble in alkali water. These properties are
essential during metal extractions or analyses. As the availability of spectrometers increased, former procedures were refined and improved, especially also with regard to the determination of toxic metals, which stays an important sub-discipline of analytical chemistry [12]. Having done much experimental work himself, Irving wrote comprehensive reviews on many aspects of dithizone chemistry [13-14]. A single mole-ratio study involving dithizone was reported by him in 1952; a mono-phase consisting of 20 % chloroform, 70 % ethanol and 10 % water was used to confirm 1M : 2L complex formation in zinc(II) dithizonate [15]. More recently, extensive structural, electrochemical [17-18], femtosecond laser spectroscopy [19-20], syntheses and photo-isomerization kinetics work [21-22] involving the dithizone ligand were reported.

After years of experience with typical related spectrometry challenges faced by students, the present chemical systems had been researched and developed, supplying a new variety of options from which the lecturer may choose. Best results are presented; Ag and Co for the mole ratio method and Ni and Co for the continuous variation method. The larger pool from which the selection was made may be viewed under Supporting Information.

Employing dithizone may perhaps have the disadvantage of both the ligand and the product absorbing strongly in the UV-visible spectral region. However, since only changes in absorbance are measured here, interference by other absorbing species is not effecting experimental outcome. Nevertheless, where possible, absorbances of complex products are obtained in non-overlapping regions of spectra. In addition, absorbance data at equivalence points only, i.e. where no species are in excess, are considered for calculating molar absorptivities, concentrations and formations constants.
Although unsubstituted dithizone itself is also soluble in aqueous alkali medium, it is not nearly as soluble as that of the −COOH derivative, which latter compound also proves to be more stable in solution. With both potassium and ammonium hydroxide being considered as bases, variation in outcome is however observed. In this regard, it should be brought to mind that ammonia itself may also act as a ligand and thus interfere during complexation reactions. High base concentration on the one hand improves ligand solubility, while on the other hand decreases stability.

Particular care is taken to weigh accurately as to obtain identical M and L concentrations where needed. Preparation of the ligand solutions are done immediately prior to experiment, as to prevent concentration and thus absorbance changes that may occur over long periods of time. For the sake of convenience and consistency, both the reagent and metal solutions may be added from either two burettes or dispenser pipettes. Clean and dry glassware are of course required at all times.

**EXPERIMENTAL**

Solvents and reagents are supplied by Sigma-Aldrich and Merck and used without further purification. Distilled water, free of interfering metals, is used. A Shimadzu UV-2550 spectrophotometer and glass cuvettes is used for UV-visible spectrophotometric measurements. All measurements are within absorbance ranges that obey Beer’s law. Significant figures are used and shown in all calculations.

**Synthesis [22]**

*(p-COOH)nitroformazan*: An acidic mixture of concentrated hydrochloric acid (30 mL) and water (50 mL) is used to dissolve 4-aminobenzoic acid (8.2170 g, 60 mmol) in a 250 mL
beaker placed on a cooling stirrer (or ice bath) and kept below 0 °C. While stirring, sodium nitrite (6.2186 g, 90 mmol) is added in small portions as to prevent the temperature from rising above 0 °C, forming the creamy yellow diazo solution. This solution is then added to a well stirred solution of sodium acetate trihydrate (100 g), glacial acetic acid (60 mL) and water (40 mL) in a 500 mL beaker. (Note: Vigorously stir the salt before and while adding the water-acid mixture or vice versa, else dissolution may not be achieved.) Nitromethane (17 mL, 300 mmol) is added to the reaction mixture, stirred at room temperature until a brick red slurry is obtained, while water is periodically added as the reaction mixture thickens. The formazan is filtered off and washed with copious amounts of cold water and dried in an open fume hood or oven at 60 °C.

*(p-COOH)*nitroformazan: orange-red (9.7 g, 93 %). M.p. 210 °C, UV-vis (acetone) \(\lambda_{\text{max}} 449\) nm. *(p-COOH)*thiocarbazone: Nitroformazan (4 g, 12 mmol) is dissolved in absolute ethanol (200 mL) in a lightly stoppered Erlen Meyer flask. Ammonium sulphide (25 mL, 70 mmol, 20 %) is added and stirred at *ca* 30 °C for 30 minutes. After 20 minutes a light yellow precipitate (thiocarbazone) forms, which is filtered off in a sintered glass funnel. The funnel containing the product is placed in a clean beaker and potassium hydroxide solution (250 mL, 2 %) is poured onto the precipitate in the funnel while stirring with a spatula to aid dissolution. The yellow thiocarbazone precipitate, *(p-COOH)*H₄Dz, is hereby oxidized to the dark red potassium salt (K⁺HĐz⁻) solution.

*(p-COOH)*dithizone: Dilute HCl (250 mL, 2 %) is cooled down to < 5 °C and slowly poured into the deep orange-red dithizonate solution until the green-black dithizone, *(p-COOH)*H₂Dz, is fully precipitated. The product mixture is centrifuged (or filtered) and washed with ample amounts of cold water (< 5 °C). For purification purposes, the former base-acid steps are repeated three times, i.e. by re-dissolving the precipitate in 2% aqueous KOH solution (200
mL) followed by addition of cold dilute HCl. The product is dried in a fume hood and washed with warm DCM (100 mL × 3). (p-COOH)dithizone: black (3 g, 73 %), M.p. 199 °C, UV-vis (acetone) \( \lambda_{\text{max}} \) 452 and 634 nm.

Metal complexation: Not all metals readily react with the neutral dithizone ligand. Fast complexation is promoted by addition of base which singly deprotonates the dithizone backbone, see Scheme 1. (For Ag, Co, Ni, Pb and Hg complex syntheses and characterization data, see Supporting Information.)

Mole Ratio Method

1. \( \text{Ag} + xL \rightarrow \text{AgL}_x \)

Two standard solutions (5.1 × 10⁻⁴ M, 100.0 mL) are prepared by dissolving:

- 0.0087 g AgNO₃ (5.1 × 10⁻⁵ mole, 169.9 g/mol) in distilled water, and
- 0.0176 g L (5.10 × 10⁻⁵ mole, 344.4 g/mol) in 0.1 M NH₄OH (aq).

To each of nine 50.0 mL volumetric flasks 2.50 mL of the silver solution is added, followed by 0.50, 1.00, 1.50, 2.00, 2.50, 3.00, 3.50, 4.00 and 4.50 mL respectively of the L solution, see Table 1. The flasks are filled to the mark with 0.1 M NH₄OH (aq). The silver concentration in all solutions is 2.6 × 10⁻⁵ M. Thirty minutes are allowed, with occasional swirling, for the reaction to complete before absorbance readings are obtained at 590 nm.

<table>
<thead>
<tr>
<th>Volume L (mL)</th>
<th>L : M</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.50</td>
<td>0.2</td>
<td>0.045</td>
</tr>
<tr>
<td>1.00</td>
<td>0.4</td>
<td>0.093</td>
</tr>
<tr>
<td>1.50</td>
<td>0.6</td>
<td>0.158</td>
</tr>
<tr>
<td>2.00</td>
<td>0.8</td>
<td>0.203</td>
</tr>
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<td>1.0</td>
<td>0.237</td>
</tr>
<tr>
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<td>0.261</td>
</tr>
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<td>3.50</td>
<td>1.4</td>
<td>0.258</td>
</tr>
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</tr>
<tr>
<td>4.50</td>
<td>1.8</td>
<td>0.263</td>
</tr>
</tbody>
</table>
Figure 1. Complexation reaction of Ag\(^+\) with (p-COOH)\(\text{H}_2\text{Dz}\): mole ratio plot of absorbance vs mole L per mole Ag\(^+\) cation, in 0.1 M NH\(_4\)OH (aq) at 590 nm. Trendlines fitted to linear sections intersect at 1M : 1L

From the mole ratio plot in Figure 1 on the x-axis, it is seen that Ag\(^+\) reacts with one ligand. This observation is expected, being consistent with the monovalent oxidation state of the silver cation.

Both Pb\(^{2+}\) and Hg\(^{2+}\) also give a 1M : 1L ratio for this particular ligand, although being divalent cations. (See Supporting Information) This could however not be confirmed by other instrumental techniques, eg. no specific ratio could be derived from mass spectra. Nevertheless, instead of the more costly silver cation, lead may be used as an example of a 1M : 1L complex.

At the equivalence point on the y-axis a difference is observed between the absorbance, \(A_{th} = 0.26\), of the theoretically fully complexed species (trendline intersect), and \(A_{exp} = 0.24\), the corresponding experimental value (curve bend). From this data the formation constant, \(K_f\), is calculated for the reaction,

\[
\text{Ag}^+ + \text{L}^- \rightleftharpoons \text{AgL}
\]

where

\[
K_f = \frac{[\text{AgL}]}{[\text{Ag}][\text{L}]} \quad - \quad (1)
\]
The experimental AgL concentration, $C_{\text{exp}}$, is calculated from the Beer law equation,

$$A = \varepsilon b C - (2)$$

where $A_{\text{th}} = 0.26$ (reaction complete), $b = 1.00 \text{ cm}$ (cuvette path length) and

$$C_{\text{th}} = \frac{5.1 \times 10^{-4} \text{ M} \times 2.50 \text{ mL}}{50.0 \text{ mL}} = 2.6 \times 10^{-5} \text{ M}$$

Thus, the molar absorptivity,

$$\varepsilon = \frac{0.26}{2.6 \times 10^{-5} \text{ M} \times 1.00 \text{ cm}} = 10 000 \text{ L mol}^{-1} \text{ cm}^{-1} \text{ at } 590 \text{ nm}$$

From equation 2

$$C_{\text{exp}} = \frac{0.24}{10 000 \text{ L mol}^{-1} \text{ cm}^{-1}} = 2.4 \times 10^{-5} \text{ M}$$

Due to the 1M : 1L reaction stoichiometry, unreacted

$$[\text{Ag}] = [\text{L}] = [\text{AgL}]_{\text{th}} - [\text{AgL}]_{\text{exp}} = 2.6 \times 10^{-5} \text{ M} - 2.4 \times 10^{-5} \text{ M} = 2.0 \times 10^{-6} \text{ M}$$

From equation 1

$$K_f = \frac{[2.4 \times 10^{-5} \text{ M}]}{[2.0 \times 10^{-6} \text{ M}][2.0 \times 10^{-6} \text{ M}]} = 6.0 \times 10^6 \text{ M}^{-1}$$

2. $\text{Co} + x\text{L} \rightarrow \text{CoL}_x$

Two standard solutions ($5.10 \times 10^{-4} \text{ M}, 100.0 \text{ mL}$) are prepared by dissolving:

- 0.0121 g CoCl$_2$·6H$_2$O ($5.10 \times 10^{-5}$ mole, 237.9 g/mol) in distilled water, and
- 0.0176 g L ($5.10 \times 10^{-5}$ mole, 344.4 g/mol) in 0.1 M KOH (aq).
To each of seven 50.0 mL volumetric flasks 2.00 mL of the cobalt solution is added, followed by 3.00, 4.00, 5.00, 6.00, 7.00, 8.00 and 9.00 mL respectively of the L solution, see Table 2. The flasks are filled to the mark with 0.1 M KOH (aq). The cobalt concentration in all solutions is $2.04 \times 10^{-5}$ M. Before absorbance readings are obtained 30 minutes are allowed, with occasional swirling, for the reaction to complete.

<table>
<thead>
<tr>
<th>Volume L (mL)</th>
<th>L : M</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.00</td>
<td>1.5</td>
<td>0.263</td>
</tr>
<tr>
<td>4.00</td>
<td>2.0</td>
<td>0.358</td>
</tr>
<tr>
<td>5.00</td>
<td>2.5</td>
<td>0.435</td>
</tr>
<tr>
<td>6.00</td>
<td>3.0</td>
<td>0.497</td>
</tr>
<tr>
<td>7.00</td>
<td>3.5</td>
<td>0.540</td>
</tr>
<tr>
<td>8.00</td>
<td>4.0</td>
<td>0.559</td>
</tr>
<tr>
<td>9.00</td>
<td>4.5</td>
<td>0.572</td>
</tr>
</tbody>
</table>

From the mole ratio plot in Figure 2 it is seen that Co$^{2+}$ reacts with three ligands. This is unexpected, however, it was recently reported elsewhere that Co$^{2+}$ undergoes fast auto-oxidation during this reaction, yielding the Co(III) complex [23].

At reaction completion, $A_{th} = 0.520$, while observed $A_{exp} = 0.497$.

(Calculations are related to the Ag mole ratio experiment.)
\[
K_f = \frac{[\text{CoL}_3]}{[\text{Co}][\text{L}^3]} \quad \text{for the reaction}
\]

\[
\text{Co}^{2+} + 3\text{L}^- \rightleftharpoons \text{CoL}_3
\]

The theoretical concentration at reaction completion,

\[
C_{\text{th}} = 2.04 \times 10^{-5} \text{ M}, \quad \text{which gives}
\]

\[
\varepsilon = 25 \, 500 \text{ L mol}^{-1} \text{ cm}^{-1} \quad \text{at 490 nm}
\]

Thus, for the \text{CoL}_3 complex

\[
C_{\text{exp}} = 1.95 \times 10^{-5} \text{ M}
\]

The 1M : 3L reaction stoichiometry results in unreacted

\[
[\text{Co}] = \frac{1}{3}[\text{L}]
\]

\[
= [\text{CoL}_3]_{\text{th}} - [\text{CoL}_3]_{\text{exp}}
\]

\[
= 9.00 \times 10^{-7} \text{ M}
\]

Thus,

\[
K_f = \frac{[1.95 \times 10^{-5} \text{ M}]}{[9.00 \times 10^{-7} \text{ M}][3 \times 9.00 \times 10^{-7} \text{ M}^3]}
\]

\[
= 1.1 \times 10^{18} \text{ M}^3
\]

**Continuous Variation Method**

1. \( \text{Ni} + x\text{L} \rightarrow \text{NiL}_x \)

Two standard solutions (5.10 \times 10^{-4} \text{ M}, 100.0 \text{ mL}) are prepared by dissolving:

- 0.0134 g NiSO\(_4\)-6H\(_2\)O (5.10 \times 10^{-5} \text{ mole, 262.8 g/mol}) in distilled water, and

- 0.0176 g L (5.10 \times 10^{-5} \text{ mole, 344.4 g/mol}) in 0.1 M KOH (aq).

To fifteen 50.0 mL volumetric flasks 0.00, 0.20, 0.50, 1.00, 1.50, 2.00, 2.50, 3.00, 4.00, 5.00, 6.00, 7.00, 8.00, 9.00 and 10.00 mL of the nickel solution is added, and also 10.00, 9.80,
9.50, 9.00, 8.50, 8.00, 7.50, 7.00, 6.00, 5.00, 4.00, 3.00, 2.00, 1.00 and 0.00 mL of the L solution, see Table 3. The flasks are filled to the mark with 0.1 M KOH (aq). Before absorbance readings are obtained 30 minutes are allowed, with occasional swirling, for the reaction to complete.

Table 3. Mixing ratio and absorbance of nickel and ligand solution mixtures.

<table>
<thead>
<tr>
<th>Volume M (mL)</th>
<th>Volume L (mL)</th>
<th>( \frac{V_M}{V_M + V_L} )</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>10.00</td>
<td>0.00</td>
<td>0.003</td>
</tr>
<tr>
<td>0.20</td>
<td>9.80</td>
<td>0.02</td>
<td>0.046</td>
</tr>
<tr>
<td>0.50</td>
<td>9.50</td>
<td>0.05</td>
<td>0.149</td>
</tr>
<tr>
<td>1.00</td>
<td>9.00</td>
<td>0.10</td>
<td>0.297</td>
</tr>
<tr>
<td>1.50</td>
<td>8.50</td>
<td>0.15</td>
<td>0.432</td>
</tr>
<tr>
<td>2.00</td>
<td>8.00</td>
<td>0.20</td>
<td>0.546</td>
</tr>
<tr>
<td>2.50</td>
<td>7.50</td>
<td>0.25</td>
<td>0.595</td>
</tr>
<tr>
<td>3.00</td>
<td>7.00</td>
<td>0.30</td>
<td>0.606</td>
</tr>
<tr>
<td>4.00</td>
<td>6.00</td>
<td>0.40</td>
<td>0.583</td>
</tr>
<tr>
<td>5.00</td>
<td>5.00</td>
<td>0.50</td>
<td>0.521</td>
</tr>
<tr>
<td>6.00</td>
<td>4.00</td>
<td>0.60</td>
<td>0.403</td>
</tr>
<tr>
<td>7.00</td>
<td>3.00</td>
<td>0.70</td>
<td>0.281</td>
</tr>
<tr>
<td>8.00</td>
<td>2.00</td>
<td>0.80</td>
<td>0.161</td>
</tr>
<tr>
<td>9.00</td>
<td>1.00</td>
<td>0.90</td>
<td>0.054</td>
</tr>
<tr>
<td>10.00</td>
<td>0.00</td>
<td>1.00</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Figure 3. Complexation reaction of Ni\(^{2+}\) with (p-COOH)$_2$Dz: continuous variation plot of absorbance vs volume fraction, in 0.1 M KOH (aq), at 690 nm. Trendlines fitted to linear sections intersect at 1M : 3L.
From the continuous variation plot in Figure 3 it is seen that Ni\(^{2+}\) reacts with three ligands. At the equivalence point the \(V_M\) to \(V_L\) ratio is \(ca\ 2.5 : 7.5\). With both the M and L solutions having similar concentrations, the mole quantity ratio is therefore also 1 : 3.

This unexpected coordination ratio is confirmed by mass spectroscopy. Additional experiments, which goes beyond the scope of this report, indicate the stable Ni(II) oxidation state persists also in the complex product where coordination by the third ligand is ascribed to dative covalent bonding. Unsubstituted dithizone, which does not have the added two \(–COOH\) coordination sites, reacts with \(Ni^{2+}\) to give the NiL\(_2\) complex [24]. (See Supporting Information for additional information.)

At the equivalence point in Figure 3 on the \(y\)-axis a difference is observed between the absorbance, \(A_{th} = 0.80\), of the theoretically fully complexed species (trendline intersect), and \(A_{exp} = 0.60\), the corresponding experimental value (curve bend). From this data the formation constant, \(K_f\), is calculated for the reaction,

\[
Ni^{2+} + 3L^- \rightleftharpoons K_f \rightarrow NiL_3
\]

where \(K_f = \frac{[NiL_3]}{[Ni][L]^3}\) - (1)

The experimental NiL\(_3\) concentration, \(C_{exp}\), is calculated from the Beer law equation,

\[
A = \varepsilon b C, \quad - \quad (2)
\]

where \(A_{th} = 0.80\) (reaction complete), \(b = 1.00\ cm\) (cuvette path length) and

\[
C_{th} = \frac{5.10 \times 10^{-4} \times 2.50 \times 50.0 mL}{50.0 mL} = 2.55 \times 10^{-5} \text{ M}
\]
Thus, the molar absorptivity

\[ \varepsilon = \frac{0.80}{2.55 \times 10^{-5} \text{ M} \times 1.00 \text{ cm}} \]

\[ = 31000 \text{ L mol}^{-1} \text{ cm}^{-1} \text{ at } 700 \text{ nm.} \]

From equation 2

\[ C_{\text{exp}} = \frac{0.60}{31000 \text{ L mol}^{-1} \text{ cm}^{-1}} \]

\[ = 1.9 \times 10^{-5} \text{ M} \]

The 1M : 3L reaction stoichiometry results in unreacted

\[ [\text{Ni}] = \frac{1}{3} [\text{L}] \]

\[ = [\text{NiL}_3]_{\text{th}} - [\text{NiL}_3]_{\text{exp}} \]

\[ = 2.55 \times 10^{-5} \text{ M} - 1.9 \times 10^{-5} \text{ M} \]

\[ = 6.5 \times 10^{-6} \text{ M} \]

From equation 1

\[ K_f = \frac{[1.9 \times 10^{-5} \text{ M}]}{[6.5 \times 10^{-6} \text{ M}][3 \times 6.5 \times 10^{-6} \text{ M}]^3} \]

\[ = 3.9 \times 10^{14} \text{ M}^{-3} \]

2. Co + xL → CoL_x

Two standard solutions (5.10 × 10^{-4} M, 100.0 mL) are prepared by dissolving:

- 0.0121 g CoCl_2·6H_2O (5.10 × 10^{-5} mole, 237.93 g/mol) in distilled water, and
- 0.0176 g L (5.10 × 10^{-5} mole, 344.35 g/mol) in 0.1 M NH_4OH (aq).

To thirteen 50.0 mL volumetric flasks 0.00, 0.50, 1.00, 1.50, 2.00, 3.00, 4.00, 5.00, 6.00, 7.00, 8.00, 9.00 and 10.00 mL of the cobalt solution is added, and also 10.00, 9.50, 9.00, 8.50, 8.00, 7.00, 6.00, 5.00, 4.00, 3.00, 2.00, 1.00 and 0.00 mL of the L solution, see Table 4. The flasks are filled to the mark with 0.1 M NH_4OH (aq). Before absorbance readings are obtained 30 minutes are allowed, with occasional swirling, for the reaction to complete.
Table 4. Mixing ratio and absorbance of cobalt and ligand solution mixtures.

<table>
<thead>
<tr>
<th>Volume M (mL)</th>
<th>Volume L (mL)</th>
<th>( \frac{V_M}{V_M + V_L} )</th>
<th>Absorbance</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>10.00</td>
<td>0.00</td>
<td>0.104</td>
</tr>
<tr>
<td>0.50</td>
<td>9.50</td>
<td>0.05</td>
<td>0.160</td>
</tr>
<tr>
<td>1.00</td>
<td>9.00</td>
<td>0.10</td>
<td>0.285</td>
</tr>
<tr>
<td>1.50</td>
<td>8.50</td>
<td>0.15</td>
<td>0.386</td>
</tr>
<tr>
<td>2.00</td>
<td>8.00</td>
<td>0.20</td>
<td>0.455</td>
</tr>
<tr>
<td>3.00</td>
<td>7.00</td>
<td>0.30</td>
<td>0.490</td>
</tr>
<tr>
<td>4.00</td>
<td>6.00</td>
<td>0.40</td>
<td>0.455</td>
</tr>
<tr>
<td>5.00</td>
<td>5.00</td>
<td>0.50</td>
<td>0.378</td>
</tr>
<tr>
<td>6.00</td>
<td>4.00</td>
<td>0.60</td>
<td>0.291</td>
</tr>
<tr>
<td>7.00</td>
<td>3.00</td>
<td>0.70</td>
<td>0.205</td>
</tr>
<tr>
<td>8.00</td>
<td>2.00</td>
<td>0.80</td>
<td>0.129</td>
</tr>
<tr>
<td>9.00</td>
<td>1.00</td>
<td>0.90</td>
<td>0.056</td>
</tr>
<tr>
<td>10.00</td>
<td>0.00</td>
<td>1.00</td>
<td>0.000</td>
</tr>
</tbody>
</table>

Figure 4. Complexation reaction of Co\(^{2+}\) with (p-COOH)H\(_2\)Dz: continuous variation plot of absorbance vs volume fraction, in 0.1 M NH\(_4\)OH (aq) at 600 nm. Trendlines fitted to linear sections intersect at 1M : 3L.

From the continuous variation plot in Figure 4 it is seen that, as for Ni\(^{2+}\), the Co\(^{2+}\) cation also reacts with three ligands. The ratio is determined as for Ni here above. This result is of course consistent with what was observed in the Co mole ratio experiment.
At reaction completion, $A_{th} = 0.580$, while observed $A_{exp} = 0.480$.

$$K_f = \frac{[CoL_3]}{[Co][L]^3} \text{ for the reaction}$$

$$Co^{2+} + 3L^- \rightleftharpoons CoL_3$$

The theoretical concentration at reaction completion,

$$C_{th} = 2.55 \times 10^{-5} \text{ M, and}$$

$$\varepsilon = 23\,000 \text{ L mol}^{-1} \text{ cm}^{-1} \text{ at 600 nm.}$$

Thus, for the CoL$_3$ complex

$$C_{exp} = 2.1 \times 10^{-5} \text{ M}$$

The 1M : 3L reaction stoichiometry results in unreacted

$$[Co] = \frac{1}{3}[L]$$

$$= [CoL_3]_{th} - [CoL_3]_{exp}$$

$$= 4.5 \times 10^{-6} \text{ M}$$

Thus,

$$K_f = \frac{[2.1 \times 10^{-5} \text{ M}]}{[4.5 \times 10^{-6} \text{ M}][3 \times 4.5 \times 10^{-6} \text{ M}]^3}$$

$$= 1.9 \times 10^{15} \text{ M}^{-3}$$

**CONCLUSION**

Utilization of the dithizone ligand derivitized with carboxy substituents on the para positions of the two phenyl rings provides reliable and uncomplicated alternatives for teaching undergraduate students some of the neat applications of UV-visible spectrophotometry. While not requiring the usual wet chemistry associated with other chemical systems that often prevent students from reaching the desired results for which such experiments are primarily designed, the
present system maintains focus on the physical spectroscopy technique. The use of aqueous media is an important cost incentive.

The larger formation constant of cobalt \((1.1 \times 10^{18} \text{ M}^{-3})\) resulting from the mole ratio study as compared to the continuous variation study \((1.9 \times 10^{15} \text{ M}^{-3})\), is ascribed to the different bases that were used. Ammonia itself acts as a ligand which competes with dithizone during the complexation reaction, and thus also in the present continuous variation experiment. The use of potassium hydroxide in the mole ratio experiment, on the contrary, facilitates a more complete reaction, and thus a larger constant of formation. Although performed in the presence of KOH, the formation constant of NiL3 \((3.9 \times 10^{14} \text{ M}^{-3})\) is almost one order of magnitude smaller than that of CoL3 in the presence of ammonia. \(K_f\) for AgL is \(6.0 \times 10^6 \text{ M}^{-1}\), which is the least stable complex in the present series. NH4OH is used in the Ag mole ratio experiment.

Observed metal to ligand ratio’s, i.e. AgL, CoL3 and NiL3, are all confirmed by mass spectroscopy and/or X-ray crystallography.

From a variety of combinations that were tested, the above four experiments yield best results.

**ASSOCIATED CONTENT**

**Supporting Information.**
Synthesis method and characterization data for the complexes; Co, Ni, Pb, Ag and Hg, with dithizone and *para*-carboxydithizone.

UV-visible spectra of the free ligand, \(p\)-COOH–H2Dz, in various solvents.
UV-visible overlay spectra of Co, Ni, Pb & Ag complexes.

Spectral change of the Co complex in methanol over time.
Mole ratio and continuous variation methods, photo’s, overlay spectra, data and plots for the various metal complexes, ligands and bases, as well as brief discussions or motivations where necessary. This material is available free of charge via the Internet at http://pubs.acs.org.

Supporting Information

Synthesis

Adapted method as reported in


tris-(p-COOH)dithizonatocobalt(III)

(p-COOH)H₂Dz (0.1520 g, 0.44 mmol) was dissolved in 50 mL aqueous KOH (0.5689 g, 10 mmol) solution, in a 250 mL beaker equipped with a magnetic stirrer. Cobalt(II) chloride hexahydrate, CoCl₂·6H₂O, (0.0320 g, 0.13 mmol,) in 50 mL water was added to the dithizonate solution. The reaction mixture was stirred for 1 hour at room temperature, to form a deep blue solution. Glacial acetic acid (20 mL) was added to precipitate out the brown black product, and the precipitate was recovered by using a centrifuge. Acid-base purification was repeated three times by repeatedly dissolving the precipitate in 0.1 M aqueous KOH and again precipitating it out by addition of 20 mL glacial acetic acid.

Co(p-COOH-HDz)₃: (0.1417 g, 89 %). M.p. > 360 °C. UV-vis (methanol) λmax 318 and 567 nm. δH (600 MHz, DMSO-d₆)/ppm: 7.5 – 8.2 (24H, Ar-H’s, 6 × C₆H₃).

Tris-dithizonatocobalt(III)

Dithizone, H₂Dz, (0.2520 g, 0.98 mmol) was dissolved in 10 mL aqueous KOH (0.5689 g, 10 mmol) solution in a 250 mL beaker equipped with a magnetic stirrer, then gradually increase the volume up to 50 mL with water. Cobalt(II) chloride hexahydrate, CoCl₂·6H₂O, (0.0752 g, 0.31
mmol) was added to the dithizonate solution and the resultant reaction mixture was stirred at room temperature for 1 hour. The product was extracted with 100 mL DCM and washed with 100 mL 0.1 M aqueous KOH until the aqueous layer was colorless. The extract was washed with 100 mL water and dried overnight in the fume hood.

Co(HDz)$_3$: (0.2163 g, 80 %). M.p. 175 °C, UV-vis (methanol) $\lambda_{\text{max}}$ 448 and 556. $\delta_H$ (600 MHz, CDCl$_3$)/ppm: 6.4 – 7.45 (30 H, Ar-H’s, 6 $\times$ C$_6$H$_5$), 9.49 (3 H, s, 3 $\times$ NH).

**tris-(p-COOH)dithizonatonickel(II)**

(p-COOH)H$_2$Dz (0.1514 g, 0.44 mmol) was reacted with nickel(II) sulphate hexahydrate, NiSO$_4$·6H$_2$O, (0.0364 g, 0.14 mmol).

Ni(p-COOH-HDz)$_3$: (0.1518 g, 95 %). M.p. $>$ 360 °C, UV-vis (methanol). $\lambda_{\text{max}}$ 315 and 458 nm. $\delta_H$ (600 MHz, DMSO-d$_6$)/ppm: 7.1 – 8.3 (24 H, Ar-H’s, 2 $\times$ C$_6$H$_5$).

**bis-dithizonatonickel(II)**

H$_2$Dz (0.2519 g, 0.98 mmol) and nickel(II) sulphate hexahydrate, NiSO$_4$·6H$_2$O, (0.1122 g, 0.41 mmol) were reacted.

Ni(HDz)$_2$: (0.2408 g, 86 %). M.p. 230 °C, UV-vis (acetone) $\lambda_{\text{max}}$ 279, 456, 560 and 686 nm. $\delta_H$ (600 MHz, DMSO-d$_6$)/ppm: 6.3 – 7.4 (20 H, Ar-H’s, 2 $\times$ C$_6$H$_5$).

**(p-COOH)dithizonatolead(II)**

(p-COOH)H$_2$Dz (0.1533 g, 0.45 mmol) was dissolved in 0.1 M aqueous NH$_4$OH and reacted with lead(II) acetate trihydrate, Pb(CH$_3$COO)$_2$·3H$_2$O, (0.1743 g, 0.46 mmol).

Pb(p-COOH-HDz): (0.1825 g, 74 %). M.p. $>$ 360 °C, UV-vis (methanol) $\lambda_{\text{max}}$ 300 and 507 nm. $\delta_H$ (600 MHz, DMSO-d$_6$)/ppm: 7.4 – 8.2 (8 H, Ar-H’s, 2 $\times$ C$_6$H$_5$), 10.3 (1 H, s, 2 $\times$ COOH).

**bis-dithizonatolead(II)**

H$_2$Dz (0.1282 g, 0.50 mmol) was dissolved in 0.1 M aqueous NH$_4$OH and reacted with lead(II) acetate, Pb(CH$_3$COO)$_2$·3H$_2$O, (0.0684 g, 0.26 mmol).

Pb(HDz)$_2$: (0.1454 g, 81 %). M.p. 210 °C, UV-vis (methanol) $\lambda_{\text{max}}$ 257 and 505 nm. $\delta_H$ (600 MHz, DMSO-d$_6$)/ppm: 6.9 – 7.9 (20 H, Ar-H’s, 4 $\times$ C$_6$H$_5$), 10.00 (2 H, s, 2 $\times$ NH).
(\(p\text{-COOH}\))dithizonatosilver(I)

(\(p\text{-COOH}\))H\(_2\)Dz (0.1530 g, 0.45 mmol) was dissolved in 0.1 M aqueous NH\(_4\)OH and reacted with silver nitrate, AgNO\(_3\), (0.0783 g, 0.46 mmol).

Ag(\(p\text{-COOH-HDz}\)): (0.1790 g, 88 %). M.p. 320 °C, UV-vis (methanol) \(\lambda_{max}\) 314 and 474 nm. \(\delta_H\) (600 MHz, DMSO-d\(_6\))/ppm: 7.4 – 8.1 (8H, Ar-H’s, 2 \(\times\) C\(_6\)H\(_4\)), 11.07 (1H, s, 1 \(\times\) NH), 12.6 (2H, s, 2 \(\times\) COOH).

dithizonatosilver(I)

H\(_2\)Dz (0.1302 g, 0.5 mmol) and silver nitrate, AgNO\(_3\), (0.0867 g, 0.51 mmol) were reacted. Silverdithizonate: (0.1700 g, 94 %). M.p. 160 °C, UV-vis (methanol) \(\lambda_{max}\) 253 and 490 nm nm. \(\delta_H\) (600 MHz, DMSO-d\(_6\))/ppm: 6.9 – 7.8 (10H, Ar-H’s, 2 \(\times\) C\(_6\)H\(_5\)), 10.38 (1H, s, 1 \(\times\) NH).

(\(p\text{-COOH}\))dithizonatophenylmercury(II)

PhHg(\(p\text{-COOH-HDz}\)): dark red (0.9678 g, 85 %). M.p. 280 °C, UV-vis (methanol) \(\lambda_{max}\) 293 and 485 nm. \(\delta_H\) (600 MHz, DMSO-d\(_6\))/ppm: 7.2 – 8.1 (13H, Ar-H’s, 1 \(\times\) C\(_6\)H\(_5\) and 2 \(\times\) C\(_6\)H\(_4\)), 11.0 (1H, s, 2 \(\times\) NH). Product photochromic.

Figure SI-1. 3.23 x 10\(^{-5}\) M \(p\text{-COOH-HDz}\) in solvents with dielectric constants above 20.
It should be noted that p-COOH–H2Dz is, amongst others, solvatochromic, concentratochromic and halochromic (pH dependant).

**NB** - Care must therefore be taken when solvents or concentrations, etc. are altered, as absorbance spectra may change completely and thus affect experimental outcomes.

**Figure SI-2.** Comparative UV-vis spectra of all metal (Co, Ni, Pb and Ag)HDz (left) and p-COOH-HDz complexes (right) in methanol. I – Co(HDz)₃, II – Pb(HDz)₂, III – Ag(HDz), IV – Ni(HDz)₂ +NH₃ and the same for p-COOH-HDz complexes (right).

UV-vis spectra of Co(HDz)₃ revealed that there may be some reaction between the complex and methanol, as the absorption maximum is initially only at 550 nm. After some time a second maximum appears at 450 nm. It remains unclear as to what reaction takes place. The change in spectra over time is illustrated in Figure SI-3.
Figure S1-3. UV-vis spectra of Co(HDz)3 at 0 min, 15 min and 5 hours (left) and decay of $\lambda_{max}$ 550 nm in methanol.

**MOLE-RATIO**

1. **Ag(p-COOH-HDz) & Ag(HDz)**

$5.11 \times 10^{-4}$ M (p-COOH)-HDz· in 0.1 M aqueous NH₄OH reacted with an aqueous solution of $5.06 \times 10^{-4}$ M Ag⁺ from AgNO₃ (0.043 g, 2.53 x $10^{-4}$ moles). To each of the eight 50.0 mL volumetric flasks, 2.50 mL of $5.06 \times 10^{-4}$ M aqueous Ag⁺ was added and also 0.50, 1.00, 1.50, 2.00, 2.50, 3.00, 3.50 and 4.00 mL of $5.11 \times 10^{-4}$ M (p-COOH)-HDz· solution. A time of 40 minutes was allowed, with occasional swirling, for the reaction to complete and the flasks were filled to the mark with aqueous 0.1 M NH₄OH.

$5.11 \times 10^{-4}$ M HDz· in 0.1 M aqueous KOH reacted with aqueous of $5.06 \times 10^{-4}$ M Ag⁺ from AgNO₃ (0.043 g, 2.53 x $10^{-4}$ moles). To each of the seven 50.0 mL volumetric flasks, 4.00 mL of $5.09 \times 10^{-4}$ M aqueous Ag⁺ was added and also 1.00, 2.00, 3.00, 4.00, 5.00, 6.00 and 7.00 mL of the $5.11 \times 10^{-4}$ M HDz· solution. A time of 30 minutes was given, with occasional swirling, for the reaction to complete, and the flasks were filled to the mark with aqueous 0.1 M KOH.

**Ag(p-COOH-HDz) solutions**
2. \( \text{Pb}(p\text{-COOH-HDz}) \) & \( \text{Pb}(\text{HDz})_2 \)

5.11 x 10^{-4} M \( (p\text{-COO}^-)\text{-HDz}^- \) in 0.1 M of NH\textsubscript{4}OH was reacted with equimolar solution of aqueous Pb(CH\textsubscript{3}COO)\textsubscript{2} \cdot 3\text{H}\textsubscript{2}O. To each of the eight 50.0 mL volumetric flasks, 2.50 mL of the 5.11 x 10^{-4} M aqueous Pb\textsuperscript{II} solution was added, and also 0.50, 1.00, 1.50, 2.00, 2.50, 3.00, 3.50 and 4.00 mL of the 5.11 x 10^{-4} M HDz\textsuperscript{-} solution. A time of 30 minutes, with occasional swirling, was allowed for the reaction to complete, and the flasks were filled to the mark with 0.1 M NH\textsubscript{4}OH solution.

3. \( \text{Ni}(p\text{-COOH-HDz})_3 \) & \( \text{Ni}(\text{HDz})_2 \)

5.11 x 10^{-4} M \( (p\text{-COO}^-)\text{-HDz}^- \) in 0.1 M aqueous KOH was reacted with an aqueous solution of 5.11 x 10^{-4} M Pb\textsuperscript{II} from Pb(CH\textsubscript{3}COO)\textsubscript{2} \cdot 3\text{H}\textsubscript{2}O (0.0121 g, 5.11 x 10^{-5} moles). To each of the seven 50.0 mL volumetric flasks, 2.00 mL of 5.11 x 10^{-4} M aqueous Pb\textsuperscript{II} was added and also 1.00, 1.50, 2.00, 2.50, 3.00, 3.50 and 4.00 mL of the 5.11 x 10^{-4} M HDz\textsuperscript{-} solution. A time of 30 minutes was given, with occasional swirling, for the reaction to complete, and the flasks were filled to the mark with aqueous 0.1 M NH\textsubscript{4}OH.

4. \( \text{Co}(p\text{-COOH-HDz})_3 \) & \( \text{Co}(\text{HDz})_3 \)

\( p\text{-COOH-HDz} \) (0.0176 g, 5.11 x 10^{-5} moles) was dissolved in \textit{ca} 1 M KOH (0.5626 g in 10 mL) in 100.0 mL volumetric flask and fill up to the mark with water, to obtain a stock solution of 5.11 x 10^{-4} M \( (p\text{-COO}^-)\text{-HDz}^- \) in 0.1 M aqueous KOH solution. CoCl\textsubscript{2} \cdot 6\text{H}\textsubscript{2}O (0.0121 g, 5.1 x 10^{-5} moles) was dissolved in 0.1 M NH\textsubscript{4}OH and filled to the mark with the solution to obtain Co\textsuperscript{III} stock solution.
moles) was dissolved in 50 mL water in a 100.0 mL volumetric flask, then filled up to the mark with water to a concentration of $5.09 \times 10^{-4}$ M. To each of the seven 50.0 mL volumetric flasks, 2.00 mL of $5.09 \times 10^{-4}$ M aqueous Co$^{II}$ solution was added, and also 3.00, 4.00, 5.00, 6.00, 7.00, 8.00 and 9.00 mL of $5.11 \times 10^{-4}$ M (p-COO$^-$)-HD$z^-$ respectively. A time of 40 min, with occasional swirling, was allowed for the reaction to complete, after which the flask was filled to the mark with aqueous 0.1 M KOH.

$5.11 \times 10^{-4}$ M HD$z^-$ in 0.1 M aqueous KOH reacted with an aqueous solution of $5.09 \times 10^{-4}$ M Co$^{II}$ from CoCl$_2$·6H$_2$O (0.0121 g, $5.1 \times 10^{-5}$ moles). To each of the seven 50.0 mL volumetric flasks, 2.00 mL of $5.11 \times 10^{-4}$ M aqueous Co$^{II}$ was added and also 3.00, 4.00, 5.00, 6.00, 7.00, 8.00 and 9.00 mL of the $5.11 \times 10^{-4}$ M HD$z^-$ solution. A time of 30 minutes was given, with occasional swirling, for the reaction to complete, and the flasks were filled to the mark with aqueous 0.1 M KOH.

Co(p-COOH-HDz) solutions

5. **Hg(p-COOH-HDz)**

A solution of $5.11 \times 10^{-4}$ M (p-COO$^-$)-HD$z^-$ in 0.1 M aqueous NH$_4$OH reacted with an aqueous solution of $5.08 \times 10^{-4}$ M Hg$^{II}$ from HgCl$_2$ (0.0138 g, $5.08 \times 10^{-5}$ moles). To each of the eight 50.0 mL volumetric flasks, 2.50 mL of $5.09 \times 10^{-4}$ M aqueous Hg$^{II}$ was added and also 0.50, 1.00, 1.50, 2.00, 2.50, 3.00, 3.50 and 4.00 mL of the $5.11 \times 10^{-4}$ M (p-COO$^-$)-HD$z^-$ solution. A time of 40 minutes, with occasional swirling, was allowed for the reaction to complete and the flasks were filled to the mark with aqueous 0.1 M NH$_4$OH. This complex was not initially part of this study, but is nevertheless reported to show that (p-COO$^-$)-HD$z^-$ reacts with large metal cations in a ratio of 1:1, as was the case for Pb.

Hg(p-COOH-HDz) solutions
CONTINUOUS VARIATION

6. Ni(p-COOH-HDz)_3 & Ni(HDz)_2

The solution of 5.11 x 10^{-4} M (p-COO⁻)-HDz⁻ in 0.1 M aqueous KOH was reacted with a solution of 5.11 x 10^{-4} M of Ni^{II} from aqueous NiSO_4⋅6H_2O (5.10 x 10^{-5} moles). To each of the twelve 50.0 mL volumetric flasks, 0.00, 0.50, 1.00, 1.50, 2.00, 3.00, 4.00, 5.00, 6.00, 7.00, 8.00, 9.00, and 10.00 mL of 5.09 x 10^{-4} M aqueous Ni^{II} was added and also 10.00, 9.50, 9.00, 8.50, 8.00, 7.50, 7.00, 6.00, 5.00, 4.00, 3.00, 2.00, 1.00 and 0.00 mL of 5.11 x 10^{-4} M (p-COO⁻)-HDz⁻ solution, respectively. A time of 40 minutes, with occasional swirling, was allowed for the reaction to complete and the flasks then filled to the mark with aqueous 0.1 M KOH.

5.11 x 10^{-4} M HDz⁻ in 0.1 M aqueous NH_4OH reacted with aqueous solutions of 5.09 x 10^{-4} M Ni^{II} from AgNO_3 (0.043 g, 2.53 x 10^{-4} moles). To each of the twelve 50.0 mL volumetric flasks, 0.00, 2.00, 4.00, 6.00, 8.00, 10.00, 12.00, 14.00, 16.00, 18.00, and 20.00 mL of 5.09 x 10^{-4} M aqueous Ni^{II} was added and also 20.00, 18.00, 16.00, 14.00, 12.00, 10.00, 8.00, 6.00, 4.00, 2.00, and 0.00 mL of 5.11 x 10^{-4} M (p-COO⁻)-HDz⁻ solution, respectively. A time of 40 minutes, with occasional swirling, was allowed for the reaction to complete and the flasks then filled to the mark with 0.1 M aqueous NH_4OH.

7. Co(p-COOH-HDz)_3

The solution of 5.11 x 10^{-4} M (p-COO⁻)-HDz⁻ in 0.1 M aqueous NH_4OH was reacted with aqueous solutions of 5.09 x 10^{-4} M Co^{II} from aqueous CoCl_2⋅6H_2O (5.09 x 10^{-5} moles). To each of the thirteen 50.0 mL volumetric flasks, 0.00, 0.50, 1.00, 1.50, 2.00, 3.00, 4.00, 5.00, 6.00, 7.00, 8.00, 9.00 and 10.00 mL of 5.09 x 10^{-4} M aqueous Co^{II} was added and also 10.00, 9.50, 9.00, 8.50, 8.00, 7.50, 7.00, 6.00, 5.00, 4.00, 3.00, 2.00, 1.00 and 0.00 mL of 5.11 x 10^{-4} M (p-COO⁻)-HDz⁻ solutions, respectively. A time of 40 minutes was allowed, with occasional swirling, for the reaction to complete and the flasks were filled to the mark with aqueous 0.1 M NH_4OH.

MOLE RATIO

\[
\text{Ag(p-COOH-HDz)} \quad \text{Absorbance} \\
\text{Moles} \quad \text{Absorbance} \\
\text{Wavelength (nm)} \quad \text{Absorbance}
\]
**Figure SI-4.** Left: Plot of absorbance vs mole \((p\text{-COO})\text{-HDz}^\cdot\) ligand per mole Ag\(^{1}\) cation, in aqueous 0.1 M NH\(_4\)OH, at 590 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

**Figure SI-5.** Left: Plot of absorbance vs mole \((p\text{-COO})\text{-HDz}^\cdot\) ligand per mole Pb\(^{11}\) cation, in aqueous 0.1 M NH\(_4\)OH, at 490 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

The lead carboxylic acid dithizonate complex (1:1) is an unexpected different stoichiometry to the unsubstituted dithizonate complex (1:2). MS fails to confirm these results, neither being indicative of either 1:1, 1:2 or 1:3 complexes. This may possibly be ascribed to dissociation during the MS ionization process. The spectrophotometric tests may however be trusted by extrapolation simply because the coordination ratio’s of other metal dithizones (Ni, Co, Ag) are proven to be correct, also by other means of characterisation. All other metal dithizonates (Co, Ag) gave the same coordination number, i.e. Co(HDz)\(_3\) and Co(p-COOH-HDz)\(_3\) and also Ag(HDz) and Ag(p-COOH-HDz), with their \(^1\)H NMR yielding the expected number of aromatic protons.

**Figure SI-6.** Left: Plot of absorbance vs mole \((p\text{-COO})\text{-HDz}^\cdot\) ligand per mole Ni\(^{11}\) cation, in aqueous 0.1 M KOH at 690 nm (similar trend observed at 465 nm). Right: Overlay spectra, dashed lines correspond to data points used in mole ratio plot.
Due to the unexpected 1:3 coordination ratio found for the Ni(II) complex, this experiment was also repeated in a polar organic solvent, namely acetone, and without the presence of base. The ligand, \((p\text{-COOH})\text{H}_2\text{Dz}\) \((0.0176 \text{ g}, 5.11 \times 10^{-5} \text{ moles})\) was dissolved in acetone in a 100.0 mL volumetric flask. An ultrasonic bath was used, obtaining the \(5.11 \times 10^{-4} \text{ M} (p\text{-COOH})\text{H}_2\text{Dz}\) solution. \(\text{NiSO}_4\cdot6\text{H}_2\text{O} \ (0.0134 \text{ g}, 5.10 \times 10^{-5} \text{ moles})\) was dissolved in warm methanol in a 100.0 mL volumetric flask, then filled up to the mark to get a solution of \(5.10 \times 10^{-4} \text{ M}\). To each of the seven 50.0 mL volumetric flasks, 2.00 mL of \(5.09 \times 10^{-4} \text{ M}\) methanolic NiII solution was added and also 1.00, 2.00, 3.00, 4.00, 5.00, 6.00 and 7.00 mL of \(5.11 \times 10^{-4} \text{ M} \ (p\text{-COOH})\text{H}_2\text{Dz}\) solution respectively. A time period of 40 minutes was allowed, with occasional swirling, for the reaction to complete, and the flasks were then filled to the mark with acetone. In the absence of base (KOH or NH4OH) the nickel complex now yielded a 1:2 ratio, Ni\((p\text{-COOH})\text{H}_2\text{Dz})_2\), see Figure 3.29. Here, the COOH substituents are not deprotonated and thus not readily available for complexation. Typically here the Ni complex has three absorption peaks (465, 560 and 690 nm). Although the slopes of the two straight lines giving the combining ratio differ not much, it is nevertheless seen that the ratio of the NiII cation to \((p\text{-COO}^-)\text{H}_2\text{Dz}^-\) ligand is 1:2. Mass spectroscopy results indicate a third \((p\text{-COO}^-)\text{H}_2\text{Dz}^-\) ligand coordinated to Ni, most probably via a \(-\text{COO}^-\) phenyl substituent.

In using the unsubstituted \(\text{H}_2\text{Dz}\), a 1:2 ratio, Ni\((\text{H}_2\text{Dz})_2\), was observed in both bases, KOH and NH4OH. The role of the carboxylate anion is therefore evendent in coordination ratio’s involving nickel. The overlay spectra in Figure 3.30 has \(\lambda_{\text{max}} = 420 \text{ and } 660 \text{ nm}\) for the Ni\((\text{H}_2\text{Dz})_2\) complex. Repeatable results were only obtained at 420 and 510 nm, while absorbance data taken at 660 nm were not consistent. Also when using 0.2 M instead of 0.1 M aqueous NH4OH, the exact same spectra were observed, with the orange colour not changing; the intensity merely increases in a non-linear fashion with increase in HDz\(^-\) concentration.

\[\text{Co}(p\text{-COOH}\text{H}_2\text{Dz})_3\]

**Figure SI-7.** Left: Plot of absorbance vs mole \((p\text{-COO}^-)\text{H}_2\text{Dz}^-\) ligand per mole CoII cation, in aqueous 0.1 M KOH at 490 nm (similar trend observed at 580 nm). Right: Overlay spectra, dashed lines correspond to data points used in mole ratio plot.
Figure SI-8. Left: Plot of absorbance vs mole \( (p\text{-COO})\text{-HDz} \) ligand per mole \( \text{Co}^{II} \) cation, in aqueous 0.1 M \( \text{NH}_4\text{OH} \) at 490 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

Results shown in Figures 3.25 and 3.26 are indicative of the \( \text{Co}^{II} \) cation reacting in a ratio of 1:3 with \( (p\text{-COO})\text{-HDz} \). These results are not affected by the type of base, namely \( \text{NH}_4\text{OH} \) or KOH.

\textbf{Ag(HDz)}

Figure SI-9. Left: Plot of absorbance vs mole HDz ligand per mole Ag\( ^{I} \) cation, in aqueous 0.1 M KOH at 470 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.
Figure SI-10. Left: Plot of absorbance vs mole HDz⁻ ligand per mole Pb²⁺ cation, in aqueous 0.1 M NH₄OH at 470 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

Figure SI-11. Left: Plot of absorbance vs mole HDz⁻ ligand per mole Ni²⁺ cation, in aqueous 0.1 M KOH at 420 nm (similar trend observed at 510 nm). Right: Overlay spectra, dashed lines correspond to data points used in mole ratio plot.

Figure SI-12. Left: Plot of absorbance vs mole HDz⁻ ligand per mole Ni²⁺ cation, in aqueous 0.1 M NH₄OH at 470 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.
Figure SI-13. Left: Plot of absorbance vs mole HDz$^-$/mole Co$^{II}$ cation, in aqueous 0.1 M KOH at 570 nm (similar trend observed at 470 nm). Right: Overlay spectra, dashed lines correspond to data points used in mole ratio plot.

CONTINUOUS VARIATION

Figure SI-14. Left: Plot of corrected absorbance vs (p-COO$^-$/HDz$^-$) volume fraction of Co$^{II}$ cation, in 0.1 M NH$_4$OH, at 600 nm. Right: Overlay spectra, dashed line corresponds to data points used in continuous variation plot.
Figure SI-15. Left: Plot of corrected absorbance vs \((p\text{-COO}^-)\text{-HDz}^-\) volume fraction of Ni\(^{II}\) cation, in aqueous 0.1 M KOH, at 700 nm. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

Figure SI-16. Left: Plot of corrected absorbance vs mole HDz\(^-\) volume fraction of Ni\(^{II}\) cation, in aqueous 0.1 M NH\(_4\)OH at 630 nm with the dashed line at maximum volume ratio. Right: Overlay spectra, dashed line corresponds to data points used in mole ratio plot.

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REFERENCES


COMPARATIVE ASSESSMENT OF THE LEVELS OF SOME HEAVY METALS IN VIRGIN AND USED WATER PLASTIC BOTTLES AND SACHETS IN NIGERIA

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ABSTRACT

Plastic bottles and sachets for water packaging manufactured from 3 different factories in Minna, Niger State of Nigeria were collected and analyzed to determine the concentration of heavy metals (Mn, Fe, Cu, Zn, Co, Cd, Pb and Ni) in them. The plastics are categorized as virgin and used plastics. The plastics were first processed by dry ashing and then digested with analar grade HNO3 in accordance with CPSC-CH-E 1002-08 method. Thereafter, the samples were quantitatively analyzed using AAS. The total mean and the standard deviation of the concentration of Mn, Fe, Cu, Zn, Co, and Ni in all the virgin plastic bottles are given as: 18.3±0.86 μg/g, 333.3±1.47μg/g, 8.3±0.68 μg/g, 889.2±1.98μg/g, 72.0±0.10μg/g and 2.5±0.10 μg/g, respectively. The total mean and the standard deviation of the levels of Mn, Fe, Cu, Zn, and Pb in the virgin plastic sachets are: 53.3±1.19μg/g, 1150.8±3.08μg/g, 206.7±2.80μg/g, 1048.3±1.91μg/g and 630.0±0.83μg/g, respectively. Cd and Pb were not detected in the virgin bottles while Cd and Co were not detected in the virgin sachets. Generally, the concentrations of these metals in the virgin plastics were found to be very low when compared with their concentration in used plastics. It was also noted that the detected heavy metals have lower concentration in virgin plastic bottles than in virgin sachets. It is concluded that environmental pollution with heavy metals arising from various human activities accounted for the relatively high concentration of these heavy metals in used plastics. On the other hand, the high concentration of these metals in plastic sachets was attributed to the colorants used to provide manufacturers’ information directly on the sachets unlike plastics bottles, which bears such information on stick label. [African Journal of Chemical Education—AJCE 7(2), July 2017]
INTRODUCTION

Plastics are synthetic materials that are made up of long chain monomer linked up together to form a polymer by polymerization process [1]. The term “Plastic” connotes the property of the materials to be moulded into almost any shape, may be soft or hard, coloured or colourless as crystal [2]. Plastics products are one of the most important materials used globally today and the amount needed is still growing. The products are very attractive, durable, easy to use and long lasting; used every day in our homes to make our lives more comfortable and our work lighter. The most common uses include food and water storage and packaging, household items, toys and furniture [3].

Plastic packaging accounted for 30% of the packaging in the market today [4]. Around 70% of overall consumer packaging consumption is used for food and beverage packaging [5]. Many different types of plastics are being used as packaging materials like plastic water bottles and sachets.

Most plastic water bottles are manufactured from polyethylene terephthalate (PET), a polymer derived from oil that adds flexibility, colour, and strength to plastic [6]. It is resistant to heat, mineral oils, solvents, acids and it is impermeable to carbonation. It is strong, light, impact resistant, naturally transparent, and completely recyclable. It does not impart a taste to its contents. This is making PET the most common plastic used for packaging food and water [7]. Roughly, 80% of the PET manufactured in the United States of America ends up in Nestlé, Coke, or Pepsi containers [7].

Polyethylene (PE) is the most common plastic used for household packaging. Polyethylene resins are milky white, translucent substances derived from ethylene, which is permeable to gases, soft waxy surface, chemical resistance, stronger, and translucent. It is used to
produce grocery bags, car fuel tanks, packaging, and piping, jerry cans, toys, bottle caps, crates [1, 8, 4, 2].

Most polymers are petrochemical compounds with additive materials to give them properties of flexibility, elasticity and resistance to fracture and transparency to light [9, 4]. Hence, the final plastic material produced contains other substances like additives, manufacturing aids, and by-products from the complex polymerization reaction that were not intentionally added to the polymers [10].

The polymers used in plastics are generally nontoxic. However, they are not used in pure form. Some inorganic compounds of heavy metals are added to the polymer. Almost all commercial plastics are compounded with monomeric ingredients to improve their processing and end-use performance [11].

The additives used in plastics processing are generally not chemically bound to the polymer; as such, they can leach out of the plastics during normal use or in response to light, chewing and other environmental factors [12]. The migration of additives or contaminants from plastic food packaging to foodstuff may be categorized into three different, but inter-related, stages: diffusion within the polymer, solvation at the polymer food interface, and dispersion into bulk food [9].

This study was carried out to assess and compare the levels of some heavy metals in virgin and used water plastic bottles and sachets in Nigeria.

MATERIALS AND METHODS

As a first step, the plastics were cut to smaller pieces and then heated. The essence of heating the plastic is to soften and deform it so that they become brittle and breakable after cooling.
The plastic were ground/pulverized to a grain size with a Mechanical grinder (Model: THOMAS WILEY MILL) which is equipped with the offset hard-metal cutters and powerful drive to make it ideally suited for the preliminary size reduction and homogenizing of the plastic sample. The plastics were ground to ensure efficient extraction of the stabilizer and other additives in the plastic sample and to increase the rate of dissolution of the sample. Appropriate amount of the ground sample was taken for further analysis.

The pulverized plastic samples were ashed to break down the organic molecules in the plastic and then digested in accordance with CPSP-CH-E1002-08 Test Method [13]. Some grams of the grounded samples were transferred into pieces in a large silica crucible where it was charred on a hot plate till all the fume escaped, as reported by [14]. This is then followed by complete ashing in muffle furnace at 500 °C for 6 hours. The crucible was then taken out of the furnace and kept in desiccators for cooling. After cooling, the samples were powdered and homogenized in the silica crucible. Then 0.2g of the sample was taken in separate silica crucible for acid digestion. 5 ml of Analar grade Nitric acid was used for digestion in an open vessel. After the initial reaction of the acid and the sample were completed, the digest was filtered using a Whatman 41 filter paper to remove the insoluble particles and the solution was brought to a final volume of 50 ml with distilled deionized water. Blank sample was also prepared similarly. Glass wares, crucibles and plastic containers were washed with liquid soaps, rinsed with distilled water and soaked in 10% HNO₃ for 24 hours; cleaned with distilled deionized water and in such a manner that no contamination occurred [15].

The processed samples were quantitatively analyzed using Buck Scientific VGP 210 Flame Atomic Absorption Spectrophotometer. After every three samples analyzed using AAS, the first sample was repeated for quality check. The result from AAS was subjected to statistical analysis.
The total mean, standard deviation and range of each heavy metals was determined using Microsoft excel.

RESULTS AND DISCUSSIONS

The concentration of Mn, Fe, Cu, Zn, Co, Cd, Pb and Ni in plastic water bottles and sachets were determined in this study. The results of the level of these heavy metals in virgin and used plastic bottles are presented in Table 1 below.

Table 1: Mean and standard deviation of heavy metals concentration in virgin and used plastic bottles

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Zn</th>
<th>Co</th>
<th>Cd</th>
<th>Pb</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>SBVB</td>
<td>25.0±1.14</td>
<td>460.0±1.80</td>
<td>5.0±0.70</td>
<td>1567.5±4.11</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
</tr>
<tr>
<td>SBUB</td>
<td>27.5±2.26</td>
<td>465.0±5.00</td>
<td>15.0±0.50</td>
<td>1947.5±0.44</td>
<td>BDL</td>
<td>BDL</td>
<td>2.5±0.44</td>
<td>2.5±0.10</td>
</tr>
<tr>
<td>GAVB</td>
<td>27.5±0.89</td>
<td>207.5±1.14</td>
<td>5.0±1.08</td>
<td>1052.5±0.46</td>
<td>72.0±0.10</td>
<td>BDL</td>
<td>BDL</td>
<td>2.5±0.17</td>
</tr>
<tr>
<td>GAUB</td>
<td>55.0±0.98</td>
<td>410.0±1.66</td>
<td>20.0±0.36</td>
<td>1152.5±0.50</td>
<td>275.0±4.04</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
</tr>
<tr>
<td>SPVB</td>
<td>2.5±0.56</td>
<td>BDL</td>
<td>15.0±0.26</td>
<td>47.5±1.37</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
</tr>
<tr>
<td>SPUB</td>
<td>27.5±0.50</td>
<td>130.0±1.32</td>
<td>17.5±0.70</td>
<td>2075.0±1.00</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
</tr>
</tbody>
</table>

From Table 1, Mn, Cu, Zn and Fe (with the exception of SPVB sample), are detected in all the analyzed plastic bottles at varying concentrations. Cd and Pb are not detected in any of the virgin bottles. This is because Cd and Pb are banned from being used in commodity plastics [16]. This indicates that manufacturers of these plastic products have complied to this regulations and that Cd and Pb have been completely replaced by other nontoxic substances in plastic pellet used to manufacture water bottles [17]. Cobalt (Co) and Ni were both detected in GAVB. Co is used as pigment to impact blue or green color to materials) [18]. Thus, GAVB contains Mn, Cu, Fe, Zn,
Co and Ni at varying concentration. It is also seen from Table 1 that GAVB contains more heavy metals than the other virgin plastics (SBVB and SPVB). SPVB contains the least amount of heavy metals, with only Mn, Cu and Zn present at a very low concentration in comparison to either SBVB or GAVB. On the other hand, SBVB have the highest concentrations of Fe and Zn compared to other virgin plastics.

From the results (Table 1), it is also observed that the concentrations of the detected heavy metals are generally lower in virgin bottles than in their used counterparts. The high concentration of these heavy metals in used plastic is attributed to environmental contamination of the plastics by the respective heavy metals. Several studies have shown that heavy metals are present in the environment at different concentration and that these metals can migrate and contaminate food, water and other materials [19, 20, 21].

For the same reason, it was observed that some heavy metals which are detected in used plastic bottles are not detected in their virgin counterparts produced by the same manufacturer. Metals like Pb and Ni were not detected in any virgin bottles but are detected in used plastic at very low concentration. This implies that these metals were not intentionally added to the plastic during production but got into them as a result of contamination. This is in agreement with the study conducted by [22] on polyethylene litters. They observed that after 10 times duplicate measurement of virgin PE pellets, the quantification limit for Pb and Cr are 11mg/kg and 12mg/kg, respectively. On the contrary, the level of Pb and Cr ranged from below the quantification limit to 10,000mg/kg in PE litters. They further observed that Pb and Cr contained in PE plastic litter exceeded 100mg/kg, which is far above the level of the metals in the virgin PE.

The results of heavy metal concentration in virgin and used plastic sachets analyzed are presented in Table 2 below. From the results, it is seen that Mn, Fe, Cu and Zn are detected in all
the plastic sachets at varying concentrations while Co, Cd and Ni are not detected in any sachets because their salts are not used in the plastics. Quite unexpected is the detection of Pb in SBVS and SPVS at 2.5μg/g and 1257.5 μg/g, respectively. Pb due to its toxicity is one of the regulated heavy metals in plastics.

Table 2: Mean and standard deviation of heavy metals concentration in virgin and used plastic sachets

<table>
<thead>
<tr>
<th>Sample</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Zn</th>
<th>Co</th>
<th>Cd</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>SBVS</td>
<td>85.0±0.79</td>
<td>1772.5±4.77</td>
<td>190.0±3.00</td>
<td>740.0±1.80</td>
<td>BDL</td>
<td>BDL</td>
<td>2.5±0.44</td>
</tr>
<tr>
<td>SBUS</td>
<td>237.5±0.56</td>
<td>6102.5±1.80</td>
<td>1252.5±1.04</td>
<td>842.5±1.40</td>
<td>BDL</td>
<td>BDL</td>
<td>137.5±2.06</td>
</tr>
<tr>
<td>GAVS</td>
<td>25.0±1.12</td>
<td>657.5±3.61</td>
<td>25.0±1.15</td>
<td>1247.5±2.57</td>
<td>BDL</td>
<td>BDL</td>
<td>BDL</td>
</tr>
<tr>
<td>GAUS</td>
<td>280.0±3.60</td>
<td>10522±4.73</td>
<td>60.0±2.95</td>
<td>1455.0±117.0</td>
<td>1157.5±1.37</td>
<td>BDL</td>
<td>BDL</td>
</tr>
<tr>
<td>SPVS</td>
<td>50.0±1.67</td>
<td>1022.5±0.87</td>
<td>405.0±4.26</td>
<td>1157.5±1.37</td>
<td>BDL</td>
<td>BDL</td>
<td></td>
</tr>
<tr>
<td>SPUS</td>
<td>607.5±3.60</td>
<td>13875.0±2.19</td>
<td>2605.0±2.00</td>
<td>3475.0±7.81</td>
<td>BDL</td>
<td>BDL</td>
<td>8612.5±0.87</td>
</tr>
</tbody>
</table>

Virgin plastic sachets are most often made from polyethylene (PE), a polymer which does not require additives to soften it. PE can be made from hard to soft by modifying the hydrocarbon chain length or cross-linking [23]. Therefore, the main sources of heavy metals to virgin sachets are colorants used to print manufacturers’ useful information.

Also evident from Table 2 is that the concentrations of the detected heavy metals are relatively high in the virgin plastics. This could be as a result of the application of high amount of compound of heavy metals as pigment during the manufacture of such plastics. All plastic sachets analyzed in this work are surface painted with ink base colorant. The manufacturers of this plastic sachet do this to make the plastic more attractive to consumers and also to indicate the
manufactures’ names and addresses. The pigments used to infer colors to plastics are compounds of heavy metals [24].

It was remarked that colorful plastics contain higher concentration of heavy metals in them than non-colored plastics [25]. It was also observed that plastic toys with outer coating paints contain high amount of tested heavy metals compared to non-painted toys [12]. As reported [26], the highest concentration of Pb is found in yellow plastic, Ni in pink and purple plastic, Zn in green and purple plastics. It was stated that heavy metals in plastic are loosely bound to the surface and can easily leach out [12]. It was further observed that heavy metals could migrate from candy packages into the candy because the ink on the packages are poorly designed that they did not maintain their structural integrity [25].

The above results (Table 2) show that the concentrations of the detected heavy metals are higher in used sachet than in the virgin sachets. The used sachet contains higher concentration of heavy metals because of environmental contamination of the used plastics sachets. It is reported that heavy metals are present in the environment, in water, food, air and our daily tools and they can be absorbed or adsorbed by materials [24, 27].

The results of the total mean value and standard deviation of the detected heavy metals in both virgin bottles and virgin sachets are as shown in Table 3 below. The range of each heavy metal in the entire virgin bottles and virgin sachet is also presented in Table 3.
### Table 3: Total Mean value with standard deviation of heavy metals concentration and range of Heavy metals in virgin plastic bottles and virgin plastic sachets

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heavy Metals Concentrations in μg/g</th>
<th>Mn</th>
<th>Fe</th>
<th>Cu</th>
<th>Zn</th>
<th>Co</th>
<th>Cd</th>
<th>Pb</th>
<th>Ni</th>
</tr>
</thead>
<tbody>
<tr>
<td>V.S</td>
<td>Mn 53.3±1.19, Fe 1150.8±3.08, Cu 206.7±2.80, Zn 1048.3±1.91, Co BDL, Cd BDL, Pb 630.0±0.83, Ni BDL</td>
<td>53.3±1.19</td>
<td>1150.8±3.08</td>
<td>206.7±2.80</td>
<td>1048.3±1.91</td>
<td>BDL</td>
<td>BDL</td>
<td>630.0±0.83</td>
<td>BDL</td>
</tr>
<tr>
<td>Range</td>
<td>Mn 25.0-85.0, Fe 657.5-1772.5, Cu 25.0-405.0, Zn 740.0-1157.5, Co - , Cd - , Pb 2.5-1257.5, Ni -</td>
<td>25.0-85.0</td>
<td>657.5-1772.5</td>
<td>25.0-405.0</td>
<td>740.0-1157.5</td>
<td>-</td>
<td>-</td>
<td>2.5-1257.5</td>
<td>-</td>
</tr>
<tr>
<td>V.B</td>
<td>Mn 18.3±0.86, Fe 336.2±1.47, Cu 16.7±0.68, Zn 889.2±1.98, Co 72.0±0.10, Cd BDL, Pb BDL, Ni 2.5±0.1</td>
<td>18.3±0.86</td>
<td>336.2±1.47</td>
<td>16.7±0.68</td>
<td>889.2±1.98</td>
<td>72.0±0.10</td>
<td>BDL</td>
<td>BDL</td>
<td>2.5±0.1</td>
</tr>
<tr>
<td>Range</td>
<td>Mn 2.5-27.5, Fe 207.5-465.0, Cu 15.0-20.0, Zn 47.5-1567.5, Co - , Cd - , Pb - , Ni -</td>
<td>2.5-27.5</td>
<td>207.5-465.0</td>
<td>15.0-20.0</td>
<td>47.5-1567.5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

It is observed from the Table 3 that virgin sachet contains higher concentration of detected metals than virgin bottles. Both PET and PE are polymers that do not require the addition of stabilizer and plasticizers, which are contributors of heavy metals, to soften them or make them flexible. The high amount of heavy metals in virgin sachets is, therefore, attributed to the higher amount of colorants applied to sachets’ surfaces to make them more attractive. Since the entire analyzed sachets are painted on the surfaces with ink based paint, it is assumed that the pigments used to color the plastic sachets are compounds of heavy metals [24, 14, 26, 25].

Mn, Fe, Cu and Zn are detected in the entire plastic sachets at high concentration when compared to their concentration in plastic bottles. Among these, Fe and Zn control predominant concentration in the virgin plastics. This could be attributed to the fact that these heavy metals are the most common metals used in plastics. Seth [28] stated that FeO is the second most common pigment used in the plastic industry and that ZnS is one of the most common white pigment used in plastics. Zn is also used as catalyst during plastic production [29]. Co and Cd were not detected in any of the sachet plastic, but Pb and Ni were detected in some of the sachet plastic.
CONCLUSIONS

In this study, the following conclusions are deduced:

1. Virgin bottles contain fewer heavy metals than used bottles.
2. Virgin bottles have lower concentration of detected heavy metals compared to used plastic bottles.
3. Virgin bottles have lower concentration of detected heavy metals compared to virgin sachets.
4. Virgin sachets have lower concentration of heavy metals compared to used sachets.
5. Compounds of Pb and Cd are no longer used in production of plastic virgin bottles.
6. Compounds of Cd and Co are no longer used to produce virgin sachets.
7. Fe and Zn are detected in all the samples at concentrations above 200ug/g.
8. Compounds of Pb are still used to color some plastic sachets.
9. Plastic products manufactured from recycled plastics will have higher concentration of heavy metals than virgin plastic products.
10. Compounds of Fe and Zn are the most popular colorant used to color plastic water bottles and sachets.

REFERENCES
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