CHEM101 Report for Laboratory Exercise #5 Synthesis of Tetramethylammonium Triiodide and Tetramethylammonium Pentaiodide [1]

Using Microsoft Word, students are to insert responses in all yellow highlighted areas. It is recommended that the report be completed without changing font size, column width, row width, margins, and highlights. The completed report must be uploaded to the CHEM 101 Brightspace site as a .pdf file by the due date posted on Brightspace. All answers must be the student's work without assistance from others. Only reports which are completed using this template will be marked.

Name: Arfaz Hossain Lab Section: B12 Quad 2 Date: June 20, 2024

Evaluation of Lab Notes

The in-lab notes exhibit several strengths, including detailed documentation of quantities and procedures, such as the specific amounts of reagents and steps like dissolving in ethanol, heating, vacuum filtration, and drying. The description of the vacuum filtration setup, including the use of a Buchner funnel and secondary trap, provides clarity, ensuring the process can be followed accurately. The yields and physical description of the products are included, aiding in verifying the outcomes. The references to the laboratory manual and sources of reagents, complete with lot numbers, can also help in repeating the experiment in the future with the correct reagents and products.

To improve the repeatability of the experiment, specific details such as the exact temperature for heating and the duration for which the solution was heated could have been included. The volume of hexanes [4] and the number of washes performed during the washing step could have been specified for clarity. Providing approximate times for dissolution and each procedural step would help ensure exact replication. Including safety precautions taken during the experiment could have also improved the comprehensiveness of the procedural guidelines in the notes for future reproductions of this experiment.

The current in-lab notes can also be used for all calculations as it detailed the exact quantities of all the reagents and their product yields. The experiment involves dissolving these reagents in 12 mL of 95% ethanol with heating and stirring. The notes describe the vacuum filtration setup, including washing with hexanes and drying the crystals under vacuum for 10 minutes. The yields are $4.119 \, g$ for $N(CH_3)_4I_3$ and $1.417 \, g$ for $N(CH_3)_4I_5$, with a description of the products' appearance. These details ensure accuracy and repeatability, supporting all necessary calculations.

Abstract

By reacting iodine (I_2) with tetramethylammonium iodide (NMe₄I) in solution with control of the reaction stoichiometry, tetramethylammonium triiodide salt (Me₄NI₃) and tetramethylammonium pentaiodide salt (Me₄NI₅) were synthesized. The % yield for Me₄NI₃ was $\frac{433}{8}$, and the % yield for Me₄NI₅ was $\frac{78.1}{8}$.

Data/Results

Table 1. Experimental data and calculated values for the preparation of Me₄NI₃ and Me₄NI₅

Synthesis	Me ₄ NI ₃	Me ₄ NI ₅
NMe ₄ I (g)	0.519	0.515
I ₂ (g)	0.531	1.317
actual yield (g)	4.119	1.417
theoretical yield (g)	0.952	1.82
% yield	433%	78.1%

Discussion *Respond to the following:*

Write the balanced chemical equations for the formation of each of the products:

 $1N(CH_3)_4I + 1I_2 \rightarrow 1N(CH_3)_4I_3$ [Stoichiometric Ratio: (1:1:1)] where *Me is replaced by CH_3* $1N(CH_3)_4I + 2I_2 \rightarrow 1N(CH_3)_4I_5$ [Stoichiometric Ratio: (1:2:1)] where *Me is replaced by CH_3*

In your own words, describe the determination of the limiting reagent in each of the preparations. (max. 5 lines)

To determine the limiting reagent, we compared the moles of each reactant based on their stoichiometric ratios. **Iodine** (I_2) ^[3] is the limiting reagent because it has fewer moles than $N(CH_3)_4I$. Tetramethylammonium Iodide ($N(CH_3)_4I$) ^[2] is the limiting reagent because it has fewer moles than I_2 .

Discuss the meaning of the % yield in your own words, do not just give a formula. Comment on the % yield observed for each of the products, including the actual values (max 4 lines).

The percent yield represents the efficiency of a chemical reaction, indicating how much product was obtained compared to the theoretical maximum possible. For $N(CH_3)_4I_3$, the percent yield was extraordinarily high at 433%, suggesting either an impurity inflating the actual yield or an incomplete dissolution of $N(CH_3)_4I$. In contrast, the percent yield for $N(CH_3)_4I_5$ was 78.1%, indicating that the reaction was relatively efficient but still had some losses or incomplete conversion.

Discuss the necessary calculations for finding the theoretical and percentage yield for each synthesis [x]

Theoretical Yield Calculation for the synthesis of Tetramethylammonium triiodide, N(CH₃)₄I₃

- = (Limiting Reagent, I₂ mass (g) ÷ I₂ molar mass (g/mol)) x (Final Product, N(CH₃)₄I₃ molar mass (g/mol)
- = (0.531 g ÷ 253.80894 g/mol) x 454.8458 g/mol
- = 0.952 (Three Significant Figures)

Percentage Yield Calculation for the synthesis of Tetramethylammonium triiodide, N(CH₃)₄I₃

- = (Experimental or actual yield of N(CH₃)₄|₃) ÷ (Calculated % yield of N(CH₃)₄|₃) x 100%
- = 4.119 g ÷ 0.952 g x 100%
- = 433% (Three Significant Figures)

Theoretical Yield Calculation for the synthesis of Tetramethylammonium pentaiodide, N(CH₃)₄I₅

- (Limiting Reagent, N(CH₃)₄I₅ mass (g) ÷ molar mass (g/mol)) x (N(CH₃)₄I₅ molar mass (g/mol)
- = (0.515 g ÷ 201.0458 g/mol) x 708.6458 g/mol
- = 1.82 (Three Significant Figures)

Percentage Yield Calculation for the synthesis of Tetramethylammonium pentaiodide, N(CH₃)₄I₅

- = (Experimental or actual yield of N(CH₃)₄I₅) ÷ (Calculated % yield of N(CH₃)₄I₅) x 100%
- = 1.417 g ÷ 1.82 g x 100%
- = 78.1 % (Three Significant Figures)

Provide the limiting reagent for each of the preparations.

For Tetramethylammonium triiodide, $N(CH_3)_4I_3$ synthesis, the limiting reagent is I_2 due to I_2 having a lower number of moles than $N(CH_3)_4I$. For Tetramethylammonium pentaiodide, $N(CH_3)_4I_5$ synthesis, the limiting reagent is $N(CH_3)_4I$ due to $N(CH_3)_4I_5$ having a lower number of moles than I_2 .

Conclusions

See page 12 of the lab manual on how to write the conclusions. (max. 2 lines)

The synthesis of $N(CH_3)_4I_3$ and $N(CH_3)_4I_5$ yielded 433% and 78.1%, respectively. The inflated %yield in the $N(CH_3)_4I_3$ synthesis could be due to incomplete dissolution of $N(CH_3)_4I$. The relatively efficient %yield for $N(CH_3)_4I_5$ synthesis indicates some losses or incomplete conversion.

References

See page 12 of the lab manual on how to format references. Do not forget to cite in the text.

- 1. Reimer, M. et al, *Laboratory Manual, Chemistry 101*, pp.35-38. (University of Victoria: Victoria, B.C.). **Summer 2024**.
- 2. Tetramethylammonium iodide. Aldrich, 6000 N Teutonia Ave, Milwaukee, WI 53209, Lot #MKCF7632.
- 3. Iodine. Bio Basic Inc., 20 Konrad Crescent, Markham, ON L3R 8T4, Lot #N9812260.

4. Hexane. Anachemia, 12000 Trans-Canada Hwy, Montreal, QC H9B 3H7, Lot #23H1761046.

Feedback Summary	max.	
Pre-lab quiz: Are all responses correct?		
Laboratory Notebook: Have all data, observations, and procedures been	1	
recorded?		
Report: Are all sections completed accurately? Are responses in the Discussion,	3	
correct? Does the conclusion only include the appropriate information? Are the		
References correctly formatted and cited?		
Participation: Did the student come prepared, was the time used well in the lab		
and was the student engaged in the experiment? Did the student show the email		
confirmation letter and request the TA to check their drawers for completeness		
before they left the lab?		
Performance evaluation: Did the student follow the safe practice guidelines		
throughout the whole lab period?		
Total mark	10	

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