



U. S. Department of Justice
Drug Enforcement Administration

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Washington, D.C. 20537

APR 05 2004

Mr. Marvin L. Van Haaften
Director
Governor's Office of Drug Control Policy
Lucas State Office Building
1st Floor
321 East 12th Street
Des Moines, Iowa 50319



Dear Mr. Van Haaften:


Enclosed is the final report containing the test results of the adulterated anhydrous ammonia provided to the DEA Special Testing and Research Laboratory (SFL1). As requested, forensic chemists at SFL1 attempted the manufacture of methamphetamine via the birch reduction (NAZI) method using the product provided mimicking clandestine manufacture in a laboratory setting. As you will see in the report, while the adulterated ammonia did not completely prevent the manufacture of methamphetamine, it did significantly limit the yield.

Please be advised that although the results of the tests conducted by SFL1 were very positive, additional testing must be conducted to determine the feasibility of obtaining the desired effect, i.e., stopping the theft of anhydrous ammonia from nursing tanks. As noted in the report, studies were not conducted by SFL1 to determine if the anhydrous ammonia could be successfully distilled from the adulterated product and subsequently used to manufacture methamphetamine. It must be noted that clandestine manufacturers have proven resourceful in the past and may not be deterred from stealing the adulterated ammonia if it can be easily distilled. Be advised that some clandestine manufacturers have resorted to synthesizing anhydrous ammonia from fertilizer utilizing a distillation process. Also, with websites devoted to the illegal manufacture of controlled substances readily available on the internet, if and when a method is devised to circumvent the adulterated product, it will be broadcast immediately.

Since these studies were conducted in a laboratory setting, it cannot be presumed that the results apply to actual clandestine manufacturing. It may prove wise to have the product tested in a field setting before reaching a final conclusion on the viability of the adulterated anhydrous ammonia for methamphetamine manufacturing. Finally, as you are most likely aware, additional studies are needed to ensure the additive is environmentally safe and does not cause damage to existing equipment, fittings, etc. which could result in injury to legitimate users and/or the general public at large.

If you have any questions regarding the report, please call Laboratory Director Jeffrey H. Comparin at (703) 668-3300 or me at (202) 307-8866.

Sincerely,


for Thomas J. Janovsky
Deputy Assistant Administrator
Office of Forensic Sciences

Enclosure

cc: Mr. Comparin – SFL1

Memorandum

**Subject**

Results from the Testing of Adulterated Anhydrous Ammonia
Provided by the State of Iowa's Office of Drug Control Policy
(FFS: 940-04)

Date

MAR 15 2004

To

Thomas J. Janovsky
Deputy Assistant Administrator
Office of Forensic Sciences

From

Jeffrey H. Comparin
Jeffrey H. Comparin
Laboratory Director
Special Testing and Research Laboratory

Subsequent to the request from your Office, Senior Research Chemist Timothy McKibben and Forensic Chemists Joseph Bozenko, Steven Schwartz, and Thomas Duncan conducted tests with the adulterated anhydrous ammonia provided by Marvin L. Van Haaften, Director, Governor's Office of Drug Control Policy, State of Iowa. The testing consisted of completing multiple reactions with varying reaction conditions and chemical analyses of the resultant products.

As shown in Mr. McKibben's attached report, the adulterated anhydrous ammonia did not completely prevent the manufacture of methamphetamine. However, the amount of methamphetamine manufactured using the adulterated ammonia was consistently very low. Although clandestine laboratory "field-like" reaction conditions were mimicked for purposes of these tests it should be noted that no attempts were made to distill the adulterated anhydrous ammonia.

If you have any questions about the information provided please contact me at (703) 668-3300.

Attachment

REC'D: 3-16-04
SF-04-752

Memorandum



Subject

Request for Testing of Ammonia Adulterant
(FFS: 940-04)

Date

March 5, 2004

To

Lance Kvetko

Supervisory Chemist

Special Testing and Research Laboratory

From

Tim McKibben

Senior Research Chemist

Special Testing and Research Laboratory

The Special Testing and Research Laboratory (SFL1) was tasked with assisting the state of Iowa's Office of Drug Control Policy and researchers at the University of Iowa with evaluating an anhydrous ammonia product treated with calcium nitrate for its ability to prevent or thwart the manufacture of methamphetamine using Birch reduction conditions. The Birch reduction is one of the most dominant methamphetamine manufacturing methods encountered in the United States, especially in the Midwestern states. Normally, a Birch reduction involves reducing the aromatic phenyl ring to a non-aromatic cyclohexadienyl or cyclohexenyl ring system. Using pseudoephedrine as the starting precursor, these reaction conditions result in the reduction of the benzylic alcohol prior to ring reduction and forms methamphetamine along with over-reduced ring reaction products. Iowa's Office of Drug Control Policy provided background information as well as a supply of the treated ammonia containing the additive and dispensing equipment. SFL1 provided the chemicals, manpower, analysis, and time to further evaluate the efficacy of this ammonia formulation.

Experiments were set up to simulate the typical "ammonia" cook seen in field conditions. Under typical conditions, the anhydrous ammonia is stolen from a nursing tank and used immediately or stored in unapproved containers such as propane tanks. The most common source of lithium metal is from lithium batteries purchased or stolen from suppliers (convenience stores). The lithium metal for this study was from a commercial source of lithium wire. Under field conditions, pseudoephedrine is typically extracted from stolen or purchased pseudoephedrine-containing over-the-counter (OTC) preparations. Spectrum Brand pseudoephedrine hydrochloride was used as the precursor for this study. The treated ammonia used in this study was from the Iowa State participants. The control ammonia was from SFL1 laboratory supplies.

Illicit Birch reactions are typically conducted in many different types of reaction vessels, often in plastic or glass containers. For the purposes of this study, all reactions were performed in small glass beakers. Procedures used in this study included varying the reaction conditions to determine the effect of such variables on the methamphetamine production capability using this ammonia formulation.

These reaction conditions include: the ammonia to lithium molar ratio, quenching agents, extraction

solvents, extraction procedures, order of reagent addition, additional proton sources, amount of ammonia used, and the ammonia tank position and agitation.

General Synthesis Procedure

Each reaction was conducted using approximately 20 grams of pseudoephedrine hydrochloride, lithium wire, either treated or untreated ammonia, and was performed inside a 250ml glass beaker. The more vigorous, reference reaction using non-treated ammonia had to be transferred to a 400ml beaker to avoid overflowing of the container. No additional measures were used to retain the ammonia longer than normal except for recharging several reactions with additional ammonia (those reactions are noted in table 1). All reaction conditions are listed in table 1.

Order of Addition and Additional Proton Sources

The order of addition of the precursor (pseudoephedrine hydrochloride), the reagent (lithium metal), and the solvent (ammonia) were varied to simulate different possible field manufacturing procedures. In addition to varying the order of addition, additional proton sources were added to several reactions to simulate the extraction of pseudoephedrine hydrochloride from pharmaceutical preparations using water or alcohol solvents.

Extraction Techniques

Two different extraction techniques were used to simulate the methods used in the field.

1. Multiple dry extractions with hexane – to simulate the use of hydrocarbon solvents such as Coleman Fuel. (**Method A**)
2. Multiple liquid-liquid extractions with diethyl ether/water – to simulate the use of traditional liquid-liquid extractions using starting fluid. (**Method B**)

The use of hexane and diethyl ether represent extraction solvents of different strengths. These two solvents are representative of known methamphetamine processing solvents. Prior to either extraction method being used, each reaction was quenched with a polar solvent (methanol or water) after unreacted lithium metal had been removed. Both extraction methods resulted in the “meth oil” being extracted and diluted to a known volume. In the ether-water, liquid-liquid extraction method it was necessary to dry the solution over sodium sulfate prior to final dilution.

Quantitation of Methamphetamine & Pseudoephedrine Reaction Samples

Quantitations were performed using gas chromatography/mass spectrometry (GC/MS) in order to confirm the identities of the components made during these syntheses. Multiple point calibrations of methamphetamine base and pseudoephedrine base were performed using n-butylamphetamine (NBA) as the internal standard. Area percents were calculated for all non-calibrated components (benzaldehyde, propylbenzene, 1-(1',4'-cyclohexadienyl)-2-methylaminopropane, 3,4-dimethyl-5-phenyloxazolidine, etc).

The area percents for these minor sample components are not included in this report. Linearities for methamphetamine and pseudoephedrine include concentrations found during this study and were anticipated prior to calibration of methamphetamine and pseudoephedrine standards. Methamphetamine base and pseudoephedrine base standards were prepared and calibrated using multi-point calibrations prior

to quantitation of reaction solutions. Reaction samples were analyzed in duplicate.

Each reaction was worked up, extracted multiple times, and then diluted to a known volume in volumetric glassware. Aliquots were removed and quantitated against methamphetamine base and pseudoephedrine base standards, using a structurally-related internal standard (NBA). Results of these analyses (table 2) are expressed as **percent yield of methamphetamine base** and **percent recovery of unreacted pseudoephedrine base**. Theoretical yields are based on individual quantities of pseudoephedrine hydrochloride (starting precursor) used in each reaction followed by conversion to theoretical quantities of methamphetamine base produced or pseudoephedrine base unchanged in each reaction.

Examples of individual drug standard total ion chromatograms (TIC) are illustrated in figures 1 and 2. The TIC of the Birch reaction standard (reaction #15) using untreated ammonia is illustrated in figure 8.

All reaction products were recovered and analyzed as base material with the exception of reaction # 4. Reaction #4 was isolated as the hydrochloride salt to determine the mass recovery of final product powder. Reaction #4 powder was weighed into a volumetric flask and dissolved with deionized water to a known volume. An aliquot was further prepared by basifying with hydroxide and extracting into an organic solvent to a known volume. This sample was analyzed using the same procedures and conditions as those used for the remaining reaction samples and standards.

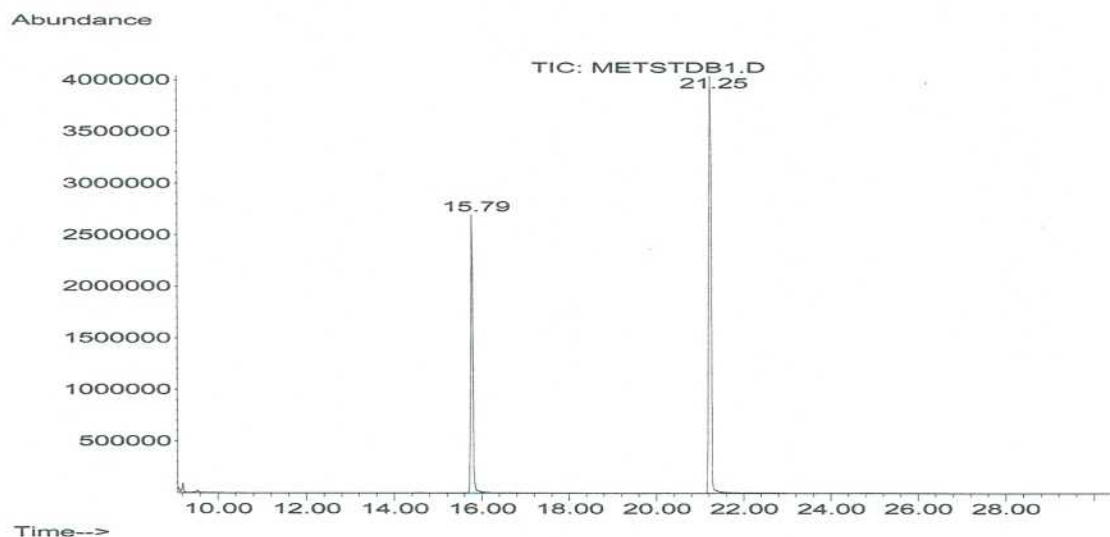


Figure 1. Methamphetamine Base Standard (15.79 min) and Internal Standard (21.25 min).

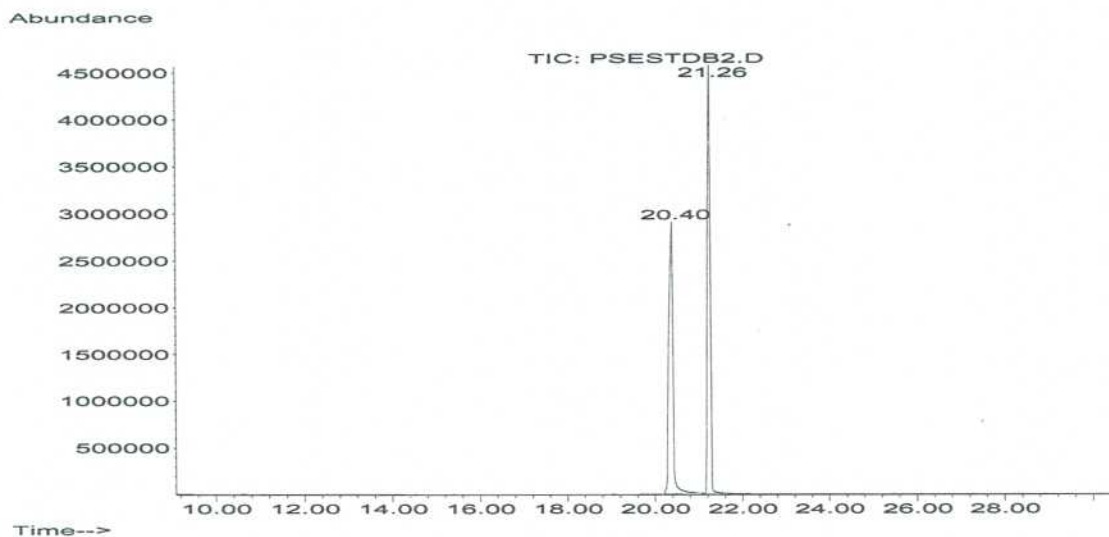


Figure 2. Pseudoephedrine Base Standard (20.40 min) and Internal Standard (21.26 min).

Inorganic Components

The calcium nitrate additive was monitored via inductively coupled mass spectrometry (ICPMS). The reaction sludge material, quantitation solutions, and pseudoephedrine were sampled for ICPMS analysis. The blue-green degradation product (caused by the treated-ammonia attacking the brass fittings on the tank adapter) was also analyzed by ICPMS.

Results and Discussion

Reaction Appearance

Anhydrous ammonia is typically observed as a clear, colorless liquid that readily refluxes at room temperature. The ammonia used in this project contained calcium nitrate as an additive. The color associated with this particular ammonia formulation was a light yellow-green as illustrated in figure 3. When the treated ammonia was allowed to evaporate overnight, the resulting material was a dark reddish-brown or rust colored liquid suspension, as seen in figure 4. Typically, Birch reduction reactions produce a well-distributed blue color formed by the ammonia-solvated electrons from lithium metal dissolution in ammonia. This blue color is seen in very dilute solutions and is persistent until the electron concentration further increases, the electrons are quenched by reaction, or the solvent evaporates. When the electron concentration increases sufficiently there is a color shift to a metallic-copper or bronze colored solution. This phenomenon is only observed in solutions of high electron concentration. Often this bronze color is seen locally near the lithium metal surface but, can also be observed throughout the reaction mixture. The typical, completely-distributed, blue color is illustrated in figure 5. The treated ammonia inhibited solvolysis of the lithium electrons and prevented the characteristic blue color from forming throughout the reaction mixture during this study. The formation of isolated pockets of blue and bronze colors being found near the surface of the lithium metal indicated that the lithium electrons were suppressed either through reaction/quenching or straight prevention of the electron solvolysis. These isolated pockets of color and known electron density are illustrated in the figures 6 and 7 for the treated-ammonia reactions.

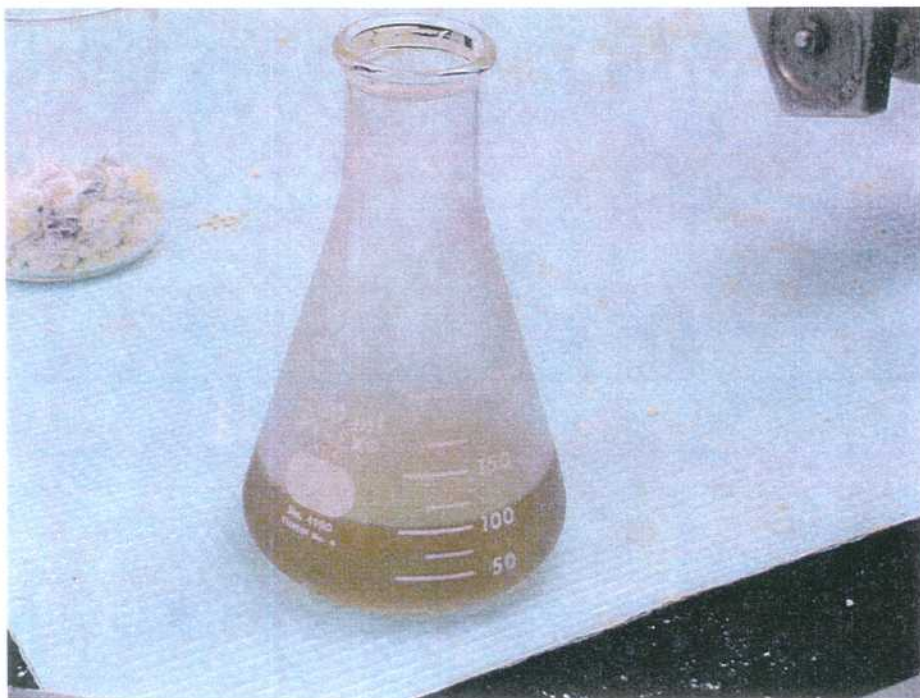


Figure 3. Treated ammonia as it comes from the tank (note the yellow-green color).

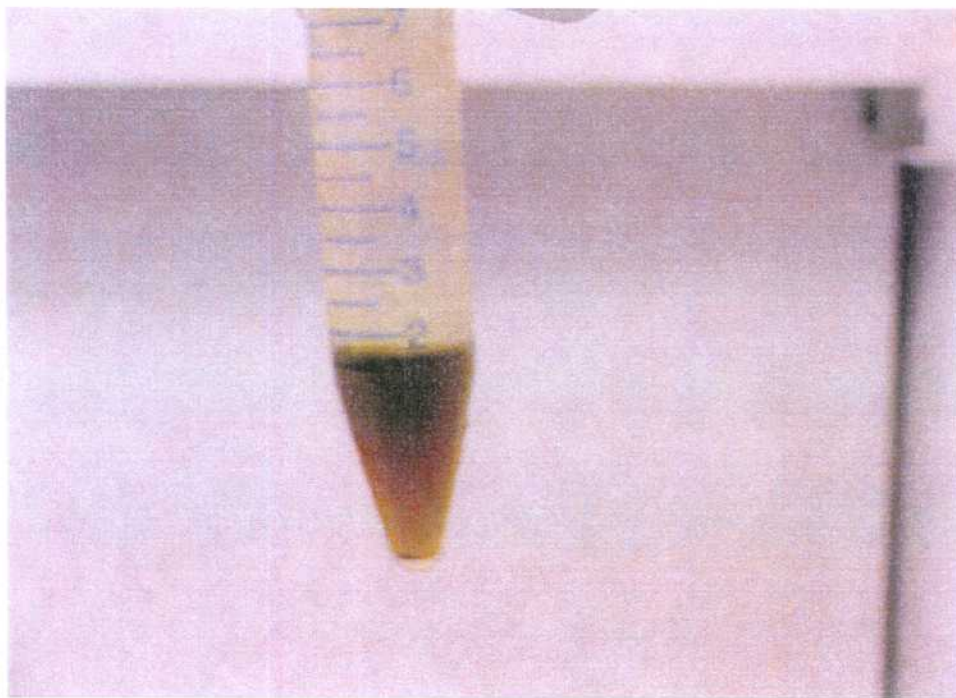


Figure 4. Remaining material after evaporation of treated-ammonia.

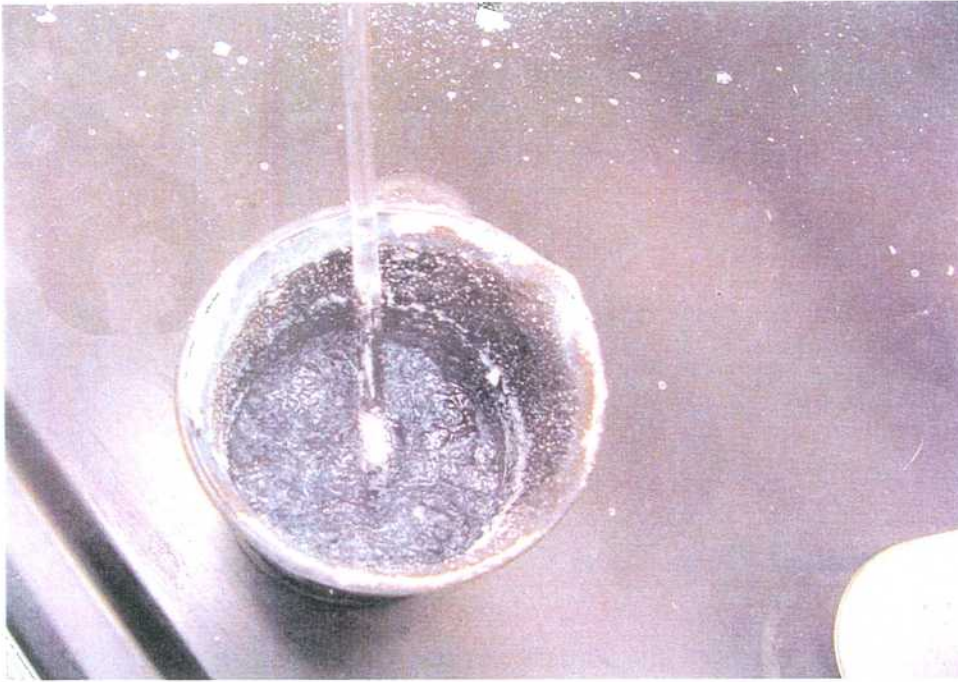


Figure 5. Typical Birch Reduction using untreated ammonia – dark blue color.



Figure 6. Birch Reduction using Iowa's treated-ammonia—No blue color in majority of reaction mixture.

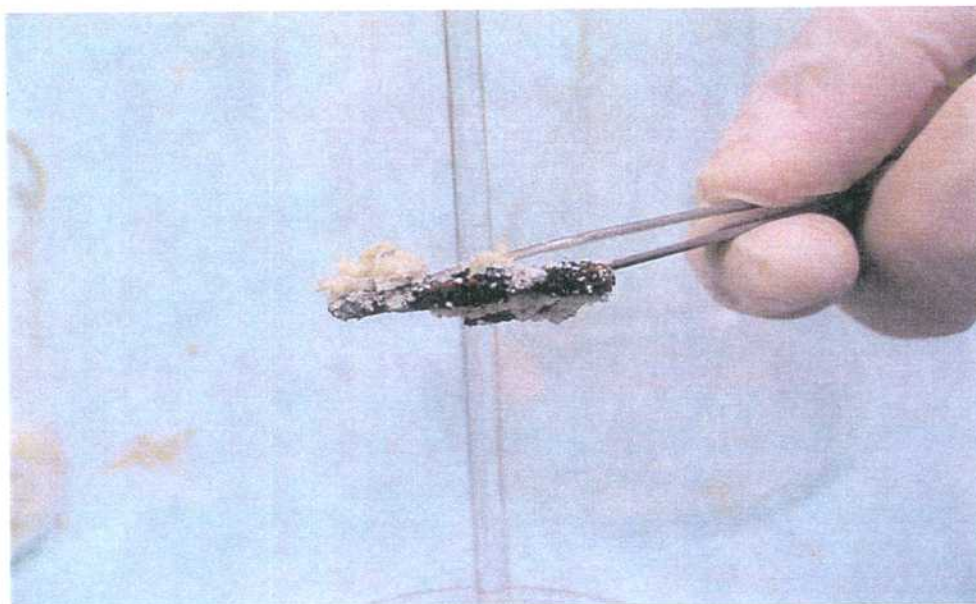


Figure 7. Bronze color on surface of unreacted Lithium metal—treated ammonia reaction.

The reaction sludge materials (insoluble material formed during reaction) recovered, using the treated-ammonia, were all more yellow in appearance than a typical Birch reaction sludge. The additive is likely acting as a sacrificial species and serves as an electron trap or sink resulting in a redox process. Sodium nitrate has been reduced to hyponitrite under sodium/mercury in liquid ammonia conditions¹. The additive may possibly be converted to hyponitrite and other species under these reaction conditions as well. It is possible that the yellow color may originate from further reduction of the hyponitrite to nitrogen dioxide, as some of the reaction sludge material was observed to change color from yellow to a white color (with only a yellow color remaining at the top of the sampling vial) upon standing. The instability of nitrite species and hyponitrite has been documented². Further transformation of the nitrogen species may also be occurring in these reactions. Further investigation of these sludges was not performed for this study.

Chemistry of the Birch Reduction

The scope of this evaluation is not specifically focused on the chemistry involved in the typical Birch reduction and will not be further expounded on in this report. The major impurity, 1-(1',4'-cyclohexadienyl)-2-methylaminopropane has been previously reported³. The reaction mechanisms for product and by-product formation are known, however, the mechanisms for inhibition using this additive requires further study and is not the focus of this report.

Table 1. Birch Reduction Reaction Conditions

RXN #	NH ₃ (ml)	Li (gm)	PSE HCL (gm)	Addition Order	Reaction Notes	Proton Source Added During Reaction	Quenching Agent and (Extraction Method)
1	50 + 50	1.40	20.01	NH ₃ /PSE/Li	NH ₃ tank slightly inverted, recharged @ 4 minutes, quenched @ 9 minutes	none	MeOH (A)
2	75	1.40	20.11	NH ₃ /Li/PSE	NH ₃ tank slightly inverted, quenched @ 9 minutes	none	MeOH (A)
3	100	1.66	20.07	NH ₃ /Li/PSE	NH ₃ tank slightly inverted, very dry material, quench @ 17 minutes	none	MeOH (A)
4	100	1.40	20.00	NH ₃ /Li/PSE	NH ₃ tank horizontal for multiple days	none	H ₂ O (A)
7	50	5.00	20.01	NH ₃ /PSE/Li	NH ₃ tank horizontal for multiple days	none	NONE (A)
9	50	1.42	20.00	NH ₃ /PSE/Li	NH ₃ tank shaken prior to cook, quenched @ 10 minutes	none	MeOH (B)
10	100 + 50	1.75	20.19	NH ₃ /Li/PSE	PSE slowly added last, RXN went dry then recharged	none	H ₂ O (B)
11	100	2.25	20.04	NH ₃ /Li/PSE	NH ₃ tank shaken and inverted prior to cook	3.46 grams of H ₂ O added to PSE prior to rxn	H ₂ O (B)
12	100	2.25	20.08	NH ₃ /Li/PSE	Allowed to get near dry prior to quench @ 12 minutes	none	H ₂ O (B)
13	110	2.01	20.03	NH ₃ /PSE/Li then remaining NH ₃ /Li added	Remaining NH ₃ /Li solution added @ 2 minutes, RXN quenched @ 20 minutes.	2.31 grams of MeOH added to PSE prior to rxn	H ₂ O (B)
14	100	1.52	20.00	Li/NH ₃ /MeOH/PSE	0.67g of MeOH added to Li/NH ₃ mixture just prior to adding solid PSE HCL	MeOH	H ₂ O (B)
15	101 + 50	1.85	20.00	Li/NH ₃ /PSE	REFERENCE BIRCH RXN	none	H ₂ O (B)

					upright tank w/ dip tube		
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All samples were given identification numbers in sequence. Non-sequential numbers are due to the sampling of ammonia, reaction sludge material, and other material between reaction sampling.

Reaction Yields and Recovery Percentages

All quantitative information is listed below in table 2. The total ion chromatogram (TIC) of the reference Birch reaction is illustrated in figure 8. Several differences were noted with the reference reaction, namely the vigorous reactivity relative to the treated-ammonia reactions and of course the typical well-distributed blue coloration from the solvated electrons. During quenching, the reference reaction also exhibited much more reactivity than did the treated-ammonia reactions. No attempts were made to optimize any of the reaction conditions and this is well illustrated by the fact that the reference reaction was incomplete. Although incomplete, a reaction yield of 42% was obtained in 12 minutes for the reference reaction. This is much greater than the methamphetamine yield for any of the other reactions using treated-ammonia. Treated ammonia reactions (TIC's) are illustrated in figures 9, 10, and 11. In general, all of these "treated" reactions produced small amounts of methamphetamine (yields range from 0.2% to 2.1%) and smaller amounts of the 1,4-cyclohexadienyl-ring impurity. The mass spectral data for this impurity is illustrated in figures 12 and 13. Small amounts of 3,4-dimethyl-5-phenyloxazolidine were observed in those reactions quenched or pretreated with methanol, and this was expected as the formaldehyde from methanol reacts with the pseudoephedrine to form the observed product. Small quantities of benzaldehyde and other components were also observed.

Pseudoephedrine recoveries were highly dependent on the extraction technique used, and ranged considerably from a low of 0% to a high of 15.7%. The use of the Method B (liquid-liquid extractions with diethyl ether) extraction technique significantly increased the amount of pseudoephedrine recovered. Recoveries ranged from a low of 0% in one reaction using Method A (direct hexane extractions) to 15.7% using Method B. However, in the Birch reference reaction, 32.4% of the unreacted pseudoephedrine was recovered, significantly more than the highest recovery in the treated ammonia reactions. Total recovery of both unreacted pseudoephedrine and manufactured methamphetamine ranged from a low of 0.6% to a high of 17.6% in the treated ammonia reactions. The highest total recoveries were among reactions using Method B as the workup procedure. The reference reaction yielded significantly more material (74.6%) than any of the treated-ammonia reactions, with Method B employed as the workup procedure.

Table 2. Quantitation Results for Treated and Untreated Birch Reductions

RXN #	% METHAMPHETAMINE REACTION YIELD	% RECOVERY OF UNREACTED PSEUDOEPHEDRINE	% TOTAL RECOVERY (PSEUDOEPHEDRINE AND METHAMPHETAMINE)
1	0.42	5.41	5.83
1	0.43	5.42	5.86
2	0.66	5.26	5.91
2	0.66	5.28	5.95
3	0.83	trace	0.83
3	0.83	trace	0.83
4	0.64	0.41	1.05

4	0.64	0.41	1.05
7	0.67	5.70	6.38
7	0.68	5.73	6.42
9	0.28	12.48	12.77
9	0.29	12.70	12.99
10	2.18	15.41	17.59
10	2.19	15.44	17.62
11	0.89	15.65	16.54
11	0.90	15.60	16.50
12	1.52	15.66	17.17
12	1.53	15.84	17.37
13	1.00	15.26	16.26
13	1.00	15.43	16.44
14	0.59	15.63	16.21
14	0.59	15.64	16.23
15	42.48	32.13	74.61
15	42.00	32.70	74.70

*Table includes individual quantitative results per reaction. Reaction # 15 is a reference Birch reduction using untreated anhydrous ammonia.

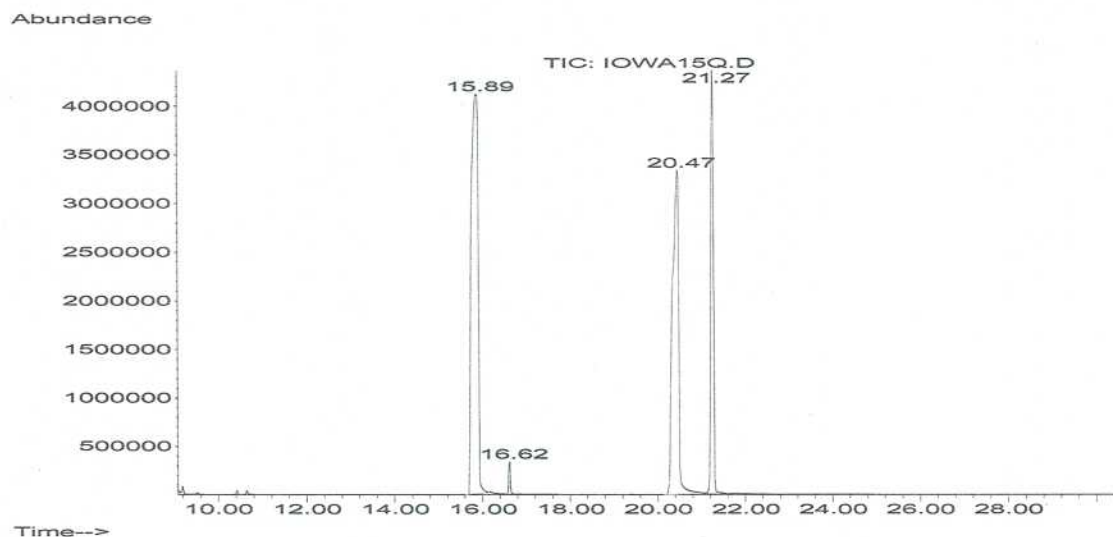


Figure 8. Reference Reaction Using Untreated Anhydrous Ammonia—methamphetamine (15.89 min), 1,4-cyclohexadienyl impurity (16.62 min), pseudoephedrine (20.47 min), and IS (21.27 min).

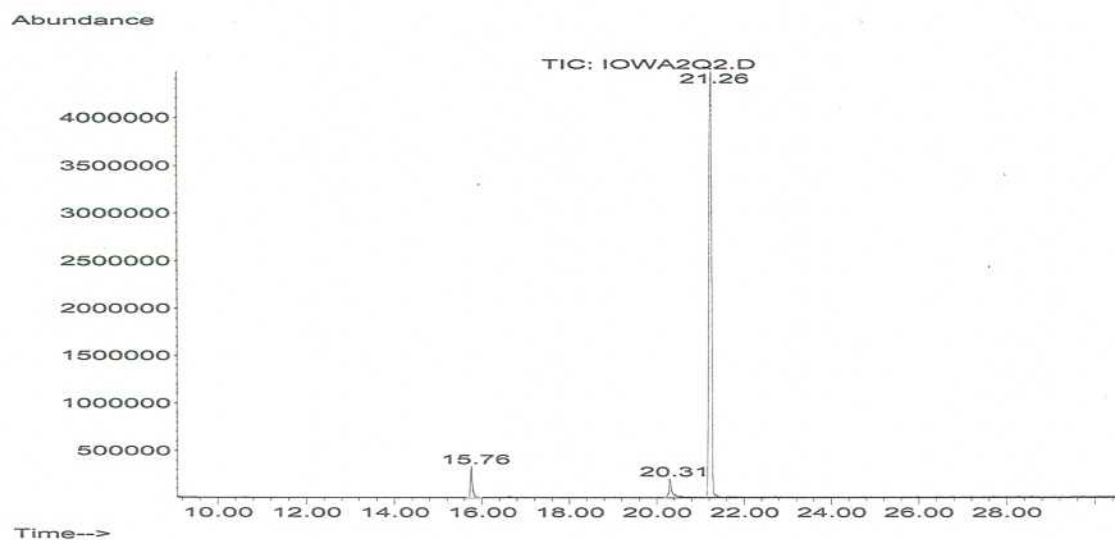


Figure 9. Birch Reduction Using Treated Ammonia, Direct Hexane Extraction (Method A)—methamphetamine (15.76 min), pseudoephedrine (20.31 min), and IS (21.26 min).

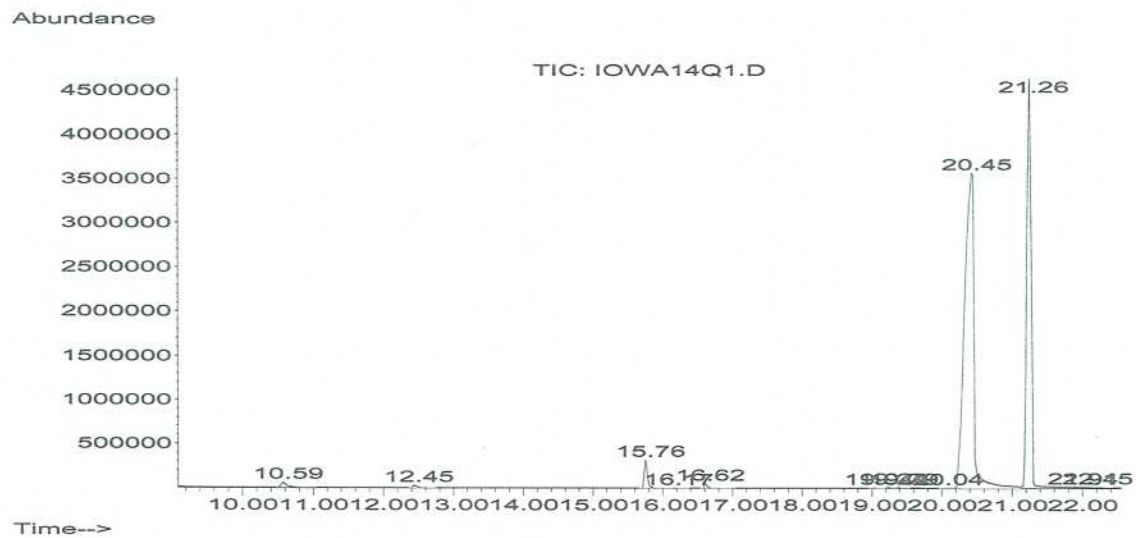


Figure 10. Birch Reduction Using Treated Ammonia, Diethyl Ether/Water Extraction (Method B)—methamphetamine (15.76 min), pseudoephedrine (20.45 min), and IS (21.26 min).

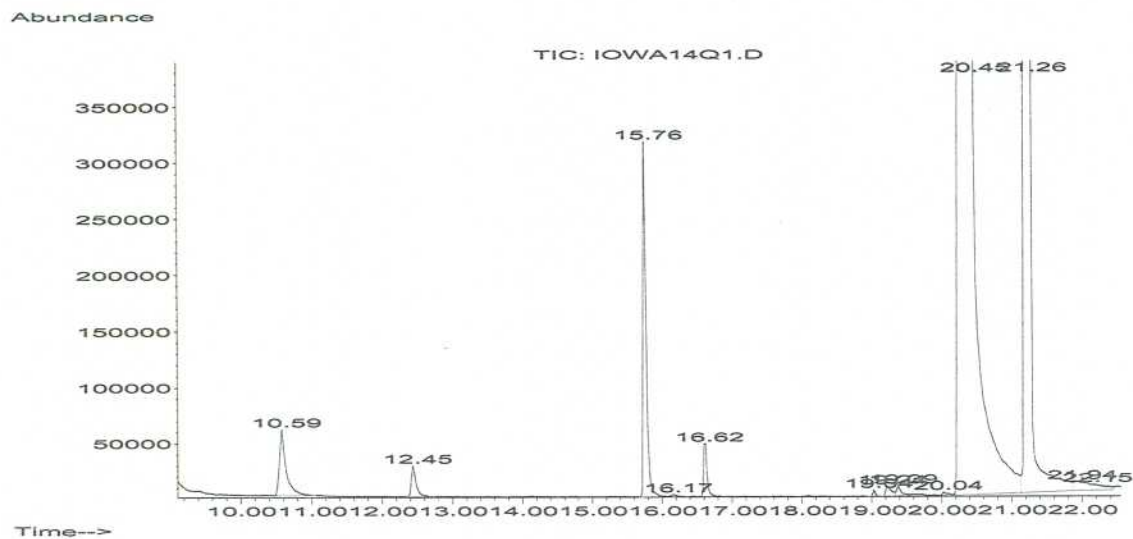


Figure 11. Birch Reduction Using Treated Ammonia, Direct Hexane Extraction (Method A), Enlarged View—methamphetamine (15.76 min), 1,4-cyclohexadienyl impurity (16.62 min), pseudoephedrine (20.42 min), and IS (21.26 min).

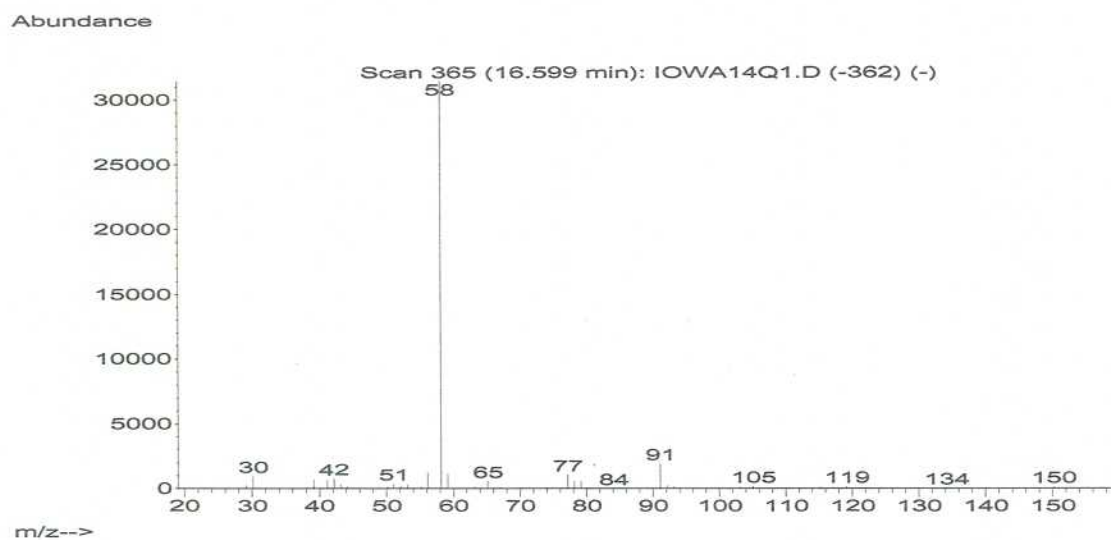


Figure 12. Birch Reduction Using Treated Ammonia—1,4-Cyclohexadienyl Impurity.

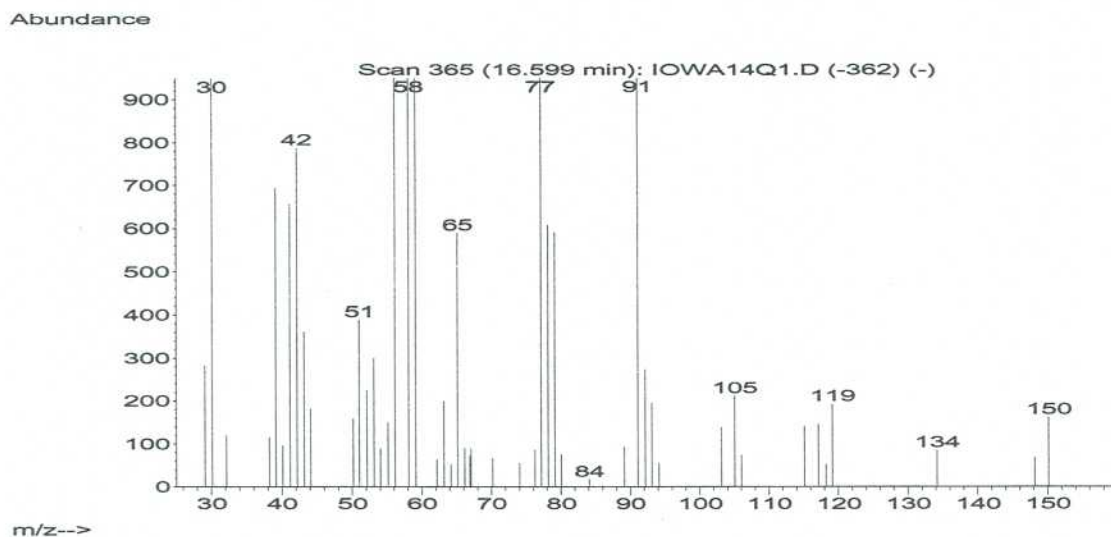


Figure 13. Birch Reduction Using Treated Ammonia—1,4-Cyclohexadienyl Impurity, Enlarged View.

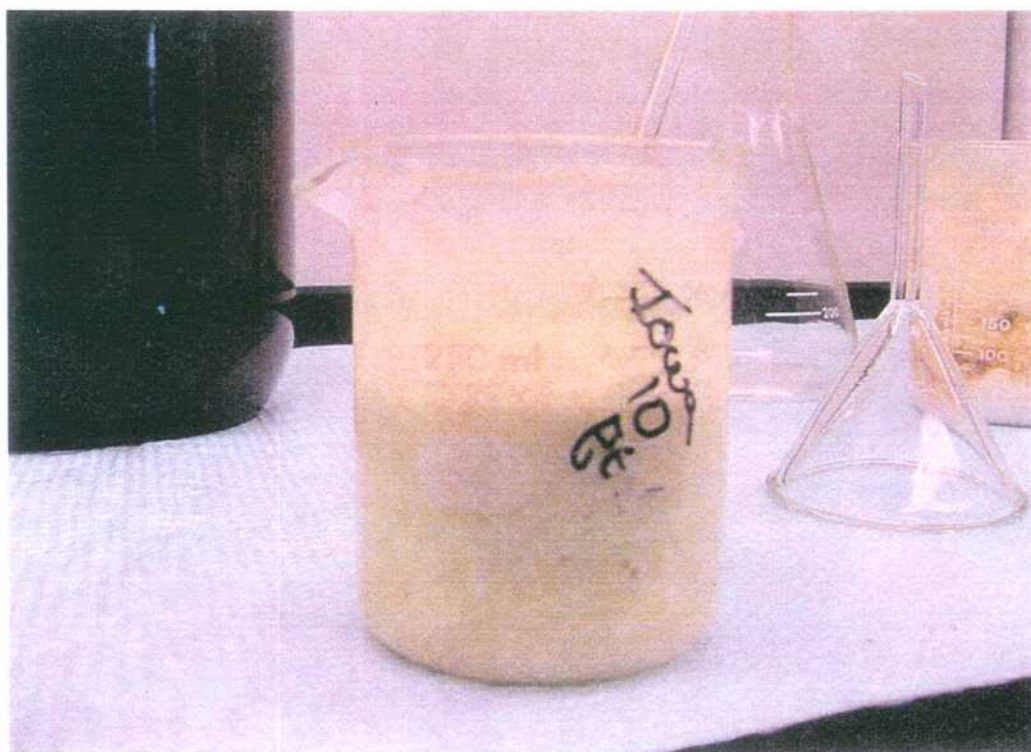


Figure 14. Iowa Reaction # 10, Ether-Water Workup – Insoluble Sludge in Ether-Water Partition.

Calcium Concentration

Initially it was observed that some of the reactions produced different responses to presumptive field tests for methamphetamine. These inconsistent field test results were thought to be caused by the additive precipitating out of the ammonia after the tank had set for some time. The reactions in question involved using the ammonia tank in different positions or handling the tank in a particular manner prior to

dispensing the ammonia into the reaction vessel. In an effort to determine if the additive was settling out of the ammonia solution upon storage or was not homogeneously distributed throughout the ammonia tank, the calcium content was monitored using Inductively Coupled Mass Spectrometry (ICPMS). Samples of treated ammonia, reaction sludge material, precursor chemical, degraded brass fitting precipitate, and reaction quantitation solutions were analyzed for calcium content. All samples were prepared for analysis by dissolving in 5.0 ml of 5% nitric acid and filtering through 0.45 micron regenerated cellulose membrane filters. The quantitation solutions were allowed to evaporate prior to dissolving in 5% nitric acid. The highest concentrations of calcium were found in the treated ammonia and the brass fitting samples (table 3). The calcium concentration in the blue amorphous material, collected from the degrading brass fitting, increased when sampled after multiple reactions on separate dates. However, the results for these particular samples were absolute concentrations and were not weight-normalized concentrations, so further study of the degraded brass material would have to be performed to obtain an exact percentage increase or decrease in the calcium concentration over time. Calcium concentrations were high (2.5 parts per million to 1.7 parts per thousand) in all reaction sludge materials but were noticeably higher in the sludge materials that were directly extracted with organic solvent. This would be expected as some of the calcium salts (calcium salts of pseudoephedrine, methamphetamine, or calcium amide) would hydrolyze when treated with water and form calcium hydroxide which is soluble in water, thus decreasing the insoluble sludge material's calcium content. The lowest calcium concentrations (13 parts per billion to 210 parts per billion) were found among the organic, quantitation solutions.

Table 3. Calcium Concentrations From Various Reaction Sources

Item Number	Description	Calcium (ppb)
IA11ANH3	Treated NH ₃ - Iowa 11 – Sampled prior to shaking	3900000
Iowa5	Treated NH ₃ - Sitting	1700000
BF213	* Brass Fitting 2/13	1600000
Iowa8	Treated NH ₃ - Shaken	1200000
Iowa4	Reaction Sludge	560000
Iowa7	Reaction Sludge	270000
BF212	* Brass Fitting 2/12	250000
Iowa1A	Reaction Sludge	250000
Iowa3	Reaction Sludge	230000
Iowa2	Reaction Sludge	140000
IA13SI2	Sludge - white bottom layer	46000
IA14SI	Reaction Sludge	11000
IA12SI	Reaction Sludge	9400
IA13SI	Sludge - orange layer at top of tube	8000
IA10SI	Reaction Sludge	7800
Iowa4FP	Finished Product	5000
IA11SI	Reaction Sludge	2500
IA10Sol	Quant Sol'n	210
IA9Sol	Quant Sol'n	140

Pseudo	Pseudoephedrine powder	100
IA13Sol	Quant Sol'n	58
IA14Sol	Quant Sol'n	58
IA12Sol	Quant Sol'n	40
IA7Sol	Quant Sol'n	39
IA3Sol	Quant Sol'n	35
IA11Sol	Quant Sol'n	35
IA1Sol	Quant Sol'n	33
IA2Sol	Quant Sol'n	13

** All concentration values listed have been normalized for sample weights except for the brass samples. Concentrations are expressed as parts per billion (ppb).*

Conclusion

The treated ammonia did not completely prevent the manufacture of methamphetamine. **However, the amount of methamphetamine actually manufactured was very low in all experiments and did not range widely. The reaction yields for methamphetamine ranged from 0.42% to 2.19%.** Only one reaction product (reaction #4) was recovered by converting the material to the final hydrochloride salt form. The total mass recovery for this reaction was only 0.2 grams. This translates to a reaction yield of 1.0% assuming the material was 100% methamphetamine hydrochloride. The powder actually was found to contain 64% methamphetamine hydrochloride. **This lowers the total yield of methamphetamine hydrochloride to 0.64%.** This result is consistent with the yield range from other reactions (reactions # 1, 2, 3, and 7) processed using similar workup procedures but isolated as the base instead of the hydrochloride powder. A single reference reaction was conducted using untreated anhydrous ammonia, the same lithium metal, and pseudoephedrine hydrochloride. A yield of 42% was observed within 12 minutes. The largest difference in yield and recoveries for these reactions was caused by the selection of a particular extraction method/solvent and not necessarily the order of reagent addition, tank position, or presence of addition proton sources. Initially there was some concern as to whether the additive may be settling out of solution and that assumption was unfounded as different handling conditions did not affect the end result of methamphetamine produced to any significant extent.

While the reference reaction was not optimized to produce the maximum amount of methamphetamine, it does illustrate a very large difference in the both quantity of methamphetamine produced (the 42% versus 2.1%) and amount of unreacted pseudoephedrine recovered (32% versus 15.7%) using the two different types of ammonia.

No attempts were made to circumvent the solid additive by distilling the treated ammonia. This would be one possible method that might be encountered in the field, to defeat the additive. The distillation of ammonia produced through the treatment of solid fertilizers (ammonium nitrate or ammonium sulfate) with base has recently become popular in certain regions of the United States.

Acknowledgements

Much of the work on this project was conducted by the following individuals, and their assistance is appreciated and should be recognized:

F/C's Joe Bozenko, Steve Schwartz, and Tom Duncan

References

1. Inorganic Chemistry, Porterfield, 1984
2. Introduction to Advanced Inorganic Chemistry, 2nd. Ed., 1970
3. Journal of the Clandestine Laboratory Investigating Chemists Association, vol. 7, #2, April 1997, pp. 7 – 10.