Phase-Transfer Catalyzed Cyclopropanation of Cyclohexene: Optimizing Dichlorocarbene Generation with TEBA

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ABSTRACT

Keywords: Carbenes; phase-transfer catalysis; [1+2] cycloadditions; cyclopropanation

Dichlorocarbene intermediates are flexible reactants, allowing for a wide variety of synthetic products. This synthesis employs phase-transfer catalysis to generate dichlorocarbene under relatively mild conditions. Sodium hydroxide and chloroform were reactants used for generating the carbene intermediate. 7,7-dichloronorcarane was produced from a carbene-cyclohexene [1+2] cycloaddition. Triethylbenzylammonium chloride was the phase-transfer catalyst (PTC) used. Mixing the aqueous and hydrophobic organic layers vigorously produced an emulsion with a large reactive interface between the phases.

The crude yield was 7.33 g with a percent yield of 45%. This was distilled under vacuum, to minimize the risk of decomposition. The purified isolate was colorless but slightly cloudy with a mass of 5.69 g, for a percent yield of 35%.

Water could have distilled over as a heteroazeotrope, so residual water, as a cause of the cloudiness had to be considered. It ruled out as there was no pronounced, broad peak observed between 3200 and 3600 cm^{-1} on IR. A very small signal on the ^{1}H NMR was observed at a shift of 7.28, where chloroform or an aromatic proton signal from TEBA would be expected. A very small peak was observed on the IR, just above 3000 cm^{-1}, indicating a possible trace of unreacted cyclohexene in the product.

1.0 INTRODUCTION

Carbenes are highly reactive and versatile intermediates that are useful in the synthesis of complex organic molecules through reactions like cyclopropanation and C-H functionalization. Carbene reactions have the reactivity of enzymatic or organometallic catalysts, generating highly electrophilic carbene intermediates.[1] Carbenes can undergo cyclopropanation of alkenes, insertion

into C–H, N–H, O–H and S–H bonds, enabling rapid assembly of complex molecular architectures under mild conditions. Among these, chloroform-derived dichlorocarbene ($:CCl_2$) is a potent electrophile, extensively employed to produce gem-dichlorocyclopropanyl structures.[2] Historically, generation techniques relied on strong bases, such as potassium tert-butoxide or sodium alkoxides, to create a carbene intermediate from chloroform. The moisture sensitive nature of these reagents required uncompromisingly inert gas and anhydrous conditions along with considerations for the significant safety risks. Also, they can generate unwanted side reactions with sensitive functional groups.[3]

The development of phase-transfer catalysis provided an alternative to these strong-base methods. Phase-transfer catalysts (PTCs) enable dichlorocarbene generation under milder conditions, allowing for reactions that wouldn't otherwise occur. Common PTCs, like quaternary ammonium or phosphonium salts, or crown ethers, will transport ionic reactants across aqueous/non-polar solvent boundaries, catalyzing carbene creation. For the PTC to be sufficiently soluble in a non-polar solvent, it needs to contain a minimum aliphatic/aromatic component, normally a minimum of thirteen carbon atoms.[4] The reaction of chloroform with aqueous sodium hydroxide directly is problematic, due to solubility issues. The use of a bridging catalyst, like a PTC, allows a hydroxide ion to migrate into the non-polar domain and react to form the dichlorocarbene intermediate.

Phase-transfer catalysis was discovered by two researchers working independently, in the mid-1960's. Micazyslaw Makosza (Poland) and Arne Brändström (Sweden) both published their work but the methodology was slow to gain widespread adoption. However, given its advantages in efficiency and green chemistry it became a bedrock technology. It was slowly recognized as a useful and efficient synthetic approach.[5]

Vacuum distillation was used to lower the distillation temperature. Many compounds are sensitive to higher temperatures, including 7,7-dichloronorcarane, which starts to decompose or undergo

ring expansion when close to its 1 atmosphere boiling point.[6] Given a compound's boiling point is defined as the temperature at which the vapor pressure matches the ambient pressure, lowering the pressure in a distillation system lowers its boiling point. This is quantitatively described, mathematically, using the Clausius-Clapeyron equation and Trouton's rule, (Equation S3, and Equation S4, shown in the 6.1 of the Supplemental Information).[7] Those equations are used to create a nomograph to determine pressure or boiling points, quickly. Figure 2 in Results and Discussion, section 2.0, illustrates this visually. Another example of reducing pressure accelerating the evaporation of a liquid is noted in a vacuum desiccator, used to speed drying of materials without the addition of heat. A complete labeled diagram for a vacuum distillation setup can be found as Figure S6, in Supplemental Information, section 6.4, Tables, Graphs, and Plots.

Benzyltriethylammonium chloride (TEBA) served as the phase-transfer catalyst, reacting aqueous NaOH with chloroform, resulting in carbene formation. The carbene is generated via an α-elimination. The resultant dichlorocarbene undergoes a [1+2] cycloaddition with cyclohexene, producing 7,7-dichloronorcarane (7,7-dichlorobicyclo[4.2.0]heptane).[8] It used solvent separation then vacuum distillation as the purification techniques.

This research was engineered to utilize a phase-transfer catalyst inducing dichlorocarbene formation in the presence of an alkene, cyclohexene, forming 7,7-dichloronorcarane, then extract and purify it via solvent extraction and vacuum distillation.

2.0 RESULTS AND DISCUSSION

The synthesis is shown in Figure 1, below:

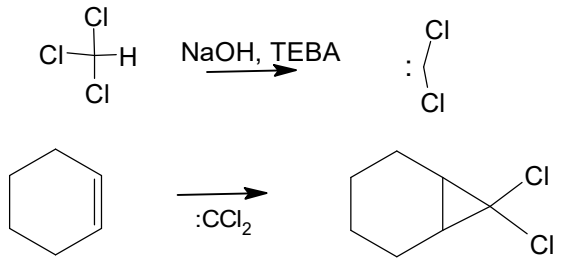

Figure 1 Synthetic path

The reaction stoichiometry was 1:1 between cyclohexene and chloroform, corresponding to a theoretical 1:1 molar ratio of cyclohexene to product. Of the reactants, cyclohexene provided the minimum number of moles, at 0.09873 mols, which predict a maximum yield of 7,7-dichloronorcarane with 0.09873 mols, at a mass of 16.3 g. The quantities of reactants and the theoretical and actual yields are summarized in Tables 1 and 2.

RMF = relative mole factor - integer

Reactants	RMF	MW	mols	mass (g)	vol (mL)	dens
Cyclohexene	1	82.14	0.0987	8.1	10	0.811
$CHCl_3$	1	119.4	0.0987	11.8	25	1.490
NaOH		39.99	0.3751	15.0		
TEBA				1.0		
CH_2Cl_2				66.3	50	1.327

Table 1 Reactions Template – Reactants

Products	*RMF*	*MW*	*mols*	*Theoretical mass (g)*	*vol (mL)*	*dens*	*Actual (crude) mass (g)*	*vol (mL)*	*% yield*	*Actual (purified) mass (g)*	*vol (mL)*	*% yield*
7,7-dichloronorcarane	*1*	*165.1*	*0.0987*	*16.30*	*12.9*	*1.26*	*7.333*	*9.2*	*45%*	*5.686*	*7.2*	*35%*

Table 2 Reactions Template – Product

The crude product was extracted via dichloromethane serial extraction, with a total of 3 aliquots used. Serial extraction leverages the exponential progression of multiple small extraction compared to the geometric scaling of a single large extraction volume, making it more efficient.

The solvent extract was then washed using brine to remove any remaining TEBA, and to help break up residual emulsion. It was dried with anhydrous Na_2SO_4 then gravity filtered. The bulk of the dichloromethane was removed using a rotary evaporator. The extract was transparent and medium brown in color. It remained open for a week for evaporation of residual dichloromethane. The crude yield dropped from 8.7 g to 7.3 g to a percent yield of 45%.

It was vacuum distilled for over an hour to purify the product. Vacuum distillation was important for 7,7-dicloronorcane purification, given at its 1 atm boiling point it could trigger autocatalytic decomposition, pyrolysis, and ring expansion. Under vacuum the product's boiling point dropped from approximately 204 °C to 143 °C. Using the Clausius-Clapeyron equation and Trouton's rule the approximate pressure used for the reduced boiling point was computed as 161 mm/Hg. Using the nomograph tool provided by Millipore-Sigma the distillation pressure was calculated at 140 mm/Hg. See nomograph diagram in Figure 2, below. Both use the Clausius-Clapeyron equation and Trouton's rule, the latter being an approximation, it can cause values to deviate from experimental values.[9]

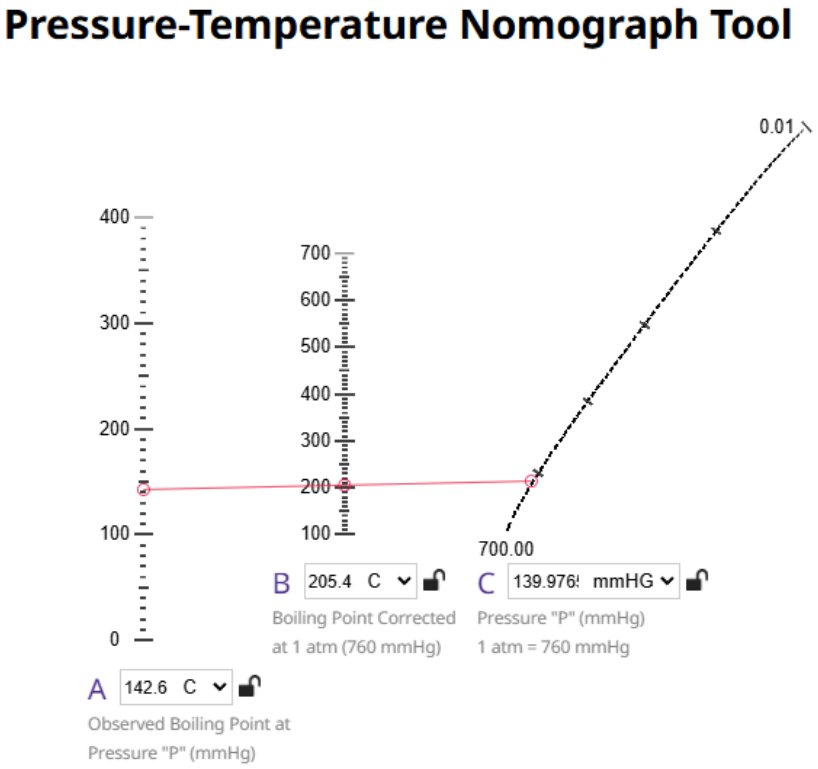

Figure 2 – Temperature/Pressure Nomograph, from Millipore Sigma (https://www.sigmaaldrich.com/US/en/support/calculators-and-apps/pressure-temperature-nomograph-interactive-tool)

The purified product was colorless, but mildly cloudy, and the yield dropped to 5.69 grams for a percent yield of 35%.

The IR showed two major C-H alkane stretching peaks which were at 2942 and 2858 cm^{-1}. There was a very small peak at or just above 3000 cm^{-1}, which may be unreacted cyclohexene. The peak was too small for the IR spectrometer to automatically list the cm^{-1}. The spectra are shown in Figures S1 (product) and S2 (cyclohexene).

The hydrogens are labeled with respect to the molecule, in Figure 3, to reference against the ^{1}H NMR peaks for ‘A’, ‘B’, and ‘C’ ({1.2 & 1.3}, 1.73, and 1.95 ppm, respectively).

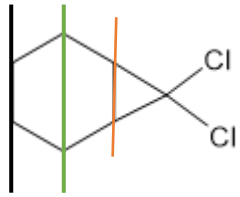

Figure 3 Hydrogen correlation figure

The chemical shifts for the 'A' hydrogen peaks are at approximately 1.2, and 1.3 ppm respectively with a total integration of 4 hydrogens. The two pairs of 'A' hydrogens are not electronically identical, given they are diastereotopic vicinal pairs. The trans hydrogens are closer to the chlorine atoms and therefore resonate at 1.3 ppm, whereas the cis hydrogens are farther from the chlorine atoms and resonate at 1.2 ppm. The 'A' hydrogens are the least deshielded by the two chlorines, so the most upfield. The 'B' hydrogens show a signal centered at 1.73 ppm with an integration of 4 hydrogens. The 'C' hydrogens peak, closest to the chlorines therefore the most deshielded, are downfield centered at 1.95 ppm with an integration at the expected 2 hydrogens. The multiplicity of the peaks was not clear due to secondary coupling effects. There was a very small signal with a chemical shift of 7.28 ppm is suspiciously close to the shift of 7.26 ppm, expected for TEBA, in $CDCl_3$.

On the ^{13}C NMR spectra, the chemical shifts of the 'A' carbons are the least deshielded, observed at 18.88 ppm, with the 'B' carbons just slightly more downfield at 20.22 ppm. The 'C', or bridge carbons present a peak at 25.82 ppm. A smaller peak is showed up at 67.44 ppm, which was expected for a cyclopropanyl carbon with two chlorines bonded to it. A large triplet signal was observed at 77.05 ppm, which is where the $CDCl_3$ signal would be expected in a ^{13}C NMR.[10]

The mass spectrum for 7,7-dichloronorcarane was performed; however, the spectrum was not specific to the product of this synthesis. It showed 4 peaks, at 165, 164, 166, and 168 m/z. The

intensities values for 164, 166, and 168 m/z fragments, respectively, are in an approximate 9:6:1 ratio. Since the natural chlorine isotopic ratio, ^{35}Cl to ^{37}Cl, is approximately 3:1, this is stochastically predictable. The 164 m/z peak corresponds to a 7,7-dichloronorcarane fragment with two ^{35}Cl atoms and ionized by the loss of a hydrogen atom. It forms the base peak (M+). Similarly, the 166 m/z fragment (M+2) has one ^{35}Cl and one ^{37}Cl with the 168 m/z fragment (M+4) has two ^{37}Cl atoms.

The lowest intensity of signal is at 165 m/z (M+1), in approximately a 1:9 ratio compared to the M+ fragment intensity. The prevalence of ^{13}C at ~1.1% of natural carbon, multiplied times seven carbons per molecule in the product, results in approximately a 1:9 ratio to the M+ fragment. ^{13}C variation in the M+ fragment is a reasonable explanation of the M+1 fragment peak.[11]

Three possible contamination sources considered were water, vacuum grease, and TEBA, resulting in cloudiness. There was a very small ^{1}H NMR signal, with a chemical shift of 7.28 ppm, possibly indicative of TEBA. A shift of 7.26 ppm is where it would be expected in $CDCl_2$.[12] Considering the peak was very small and the distillation was never vigorous; this signal alone does not appear completely sufficient nor conclusive in accounting for the observed impurity. Chloroform also has a peak at 7.26 ppm, but due to its miscibility with 7,7-dichloronocane it would not be the cause of the cloudiness.[13] The other main candidate for contamination was water, due to its ability to form a heteroazeotrope with 7,7-dichloronocarane.[14] Water contamination was eliminated because the IR had no broad, U-shaped band observed from 3200 – 3600 cm^{-1}. No 1260 cm^{-1} Si-CH_3 stretch IR signal was seen and no 800 cm^{-1} Si-C vibration / CH_3 rocking was observed, but there was a 1026 cm^{-1} peak that an Si-O-Si backbone would have produced.

3.0 EXPERIMENTAL

3.1 Chemicals

The following chemicals were used as standards for the experiments described in this paper: benzyltriethylammonium chloride – 9%, Acros Organics, Milwaukee, WI. CAS# 56-37-1; chloroform – reagent grade, stabilized with 1% V/V ethanol, Caledon Labs Ltd., Ontario, Canada. CAS# 67-66-3; cyclohexene – reagent grade > 98%, contains 0.01% tert-butylcresol, Fisher Scientific, Fair Lawn, NJ. CAS# 110-83-8; dichloromethane – ACS certified, stabilized, , Fisher Scientific, Fair Lawn, NJ. CAS# 75-09-2; sodium sulfate, anhyd. – powder, Fisher Scientific, Fair Lawn, NJ. CAS# 7757-82-6; sodium chloride – ACS grade, crystalline, , Fisher Scientific, Fair Lawn, NJ. CAS# 7647-14-5; sodium hydroxide – pellets, Fisher Scientific, Fair Lawn, NJ. CAS# 1310-73-2; and chloroform-d – for NMR, 99.6 atom% D, Fisher Scientific, Fair Lawn, NJ. CAS# 865-49-6.

3.2 Equipment

The equipment used included rotary evaporator, model Hei-VAP Core, manufactured by Heidolph Instruments GmbH & Co. KG, Walpersdorfer Str. 12, 91126 Schwabach, Germany; PerkinElmer Spectrum IR, 710 Bridgeport Avenue, Shelton, CT 06484; Bruker NMR, Bruker Avance 400 MHz, manufactured by Bruker BioSpin, 40 Manning Road, Billerica, MA 01821; and the Nicolet iS10 FTIR spectrometer, manufactured by Thermo Fisher Scientific, 2797 Peterson Pt NW, Norcross, Ga 30071.

3.3 Procedures

15 g of sodium hydroxide were dissolved in 15 mL of water, in a 250-mL Erlenmeyer flask, stirring until dissolved. It was cooled to 18 °C, using an ice bath and a stirring bar added. The flask was placed on a stirrer.

In a 100-mL Erlenmeyer flask, 10 mL of cyclohexene and 25 mL of chloroform were placed, then swirled to mix.

The cyclohexene/chloroform mixture was slowly added to the stirred sodium hydroxide solution. The stirrer was set on high. The temperature was checked periodically, manually and with a thermometer. The mixture was at 26 °C.

At approximately 30 minutes the temperature was measured at under 30 °C and the stirrer stopped. The mixture was allowed to stand for an additional 30 min and was manually swirled at 10-min intervals. 75 mL of water was added to the mixture and swirled to ensure all emulsion was incorporated, then the contents poured into a separatory funnel.

To the funnel, 25 mL of dichloromethane was added and the funnel capped. The mixture mixed gently, then the setup was tilted up and excess pressure relieved via the stopcock. The mixture was mixed vigorously for about a minute.

Once the layers separated the brown dichloromethane layer was drained into a beaker. Another 25 mL of dichloromethane was added and the extraction repeated. Again, the dichloromethane layer was drained, and another 25 mL of dichloromethane was added.

The aqueous layer was allowed to stand, the dichloromethane extracts returned to the funnel, and a brine solution was added to the funnel, to wash the solvent extract. The funnel was again sealed, the pressure equilibrated and shaken. The extract layer was emptied into its beaker and the brine was discarded. The extract was returned to the funnel and another measure of brine added, repeating the extraction with the brine. The extract was drained into its beaker and the brine was discarded.

To the beaker, two small scoops of anhydrous sodium sulfate were added and swirled. The extract was gravity-filtered through folded filter paper. The extract and dehydrating agent were poured

into the funnel to gravity filter. Once all had drained through, a small amount of dichloromethane was used to rinse the beaker and poured over the residue in the filter paper, removing the remaining product still in the dehydrating agent.

A round-bottom 100 mL flask with ground glass fitting was weighed and the weight recorded. Part of the contents of the beaker were poured into the flask, and it was attached to a Hei-VAP Core rotary evaporator (rotovap) made by Heidolph Instruments. The rotovap was used to evaporate the bulk of the dichloromethane. As the boiling subsided, remaining extract was added, repeating the procedure until the total extract had been reduced and no longer boiled. The flask was reweighed to get an initial crude yield weight.

The flask of residue was placed in a drawer so the remaining CH_2Cl_2 could evaporate. The crude material initially weighed 8.734 g, decreasing to 7.333 g after residual dichloromethane evaporated over one week. This reduced the crude yield to 54% of theoretical.

Glassware was set up for vacuum distillation with a small stirring bar, the crude product placed in the round bottom flask and heated, the thermometer placed, with the vacuum turned on and the condenser water running. The variac autotransformer was set to 40%, then increased by 10% every 10 minutes. After 30 minutes, the product was boiling without product being condensed. Aluminum foil was used to insulate the round bottom flask containing the crude product and neck of the three-way adapter, to reduce condensation prior to the vapor reaching the condenser. At 40 minutes the transformer was up to 80% and the temperature of the thermometer rising. As product started to condense in the condenser, the temperature had reached 140 °C. As more of the product distilled over, the temperature rose to 143 °C and remained at that temperature through the rest of the distillation. The boiling remained gentle through the entire distillation. At 60 minutes of distillation the crude residue appeared to be about 2 mL left, so the power was reduced to zero, the

aluminum foil insulation removed, and the remaining distillate was allowed to drain from the condenser.

The distillate was slightly cloudy. It was weighed, having a mass of 5.686 g. It was labeled compound 1. The Nicolet iS10 FTIR spectrometer did an IR scan of the purified product. About 50 mg was sent for NMR/mass spectrum.

3.4 Product Characterization

1, *7,7-dichlorobicyclo[4.2.0]heptane:* slightly cloudy, colorless liquid, yield 5.69 g, % yield 35%; IR (neat), υ/cm^{-1} 2942, 2858, 1462, 1445, 1393, 1335, 1226, 1167, 1025, 910, 842, 833, 793, 751; ^{1}H NMR (400 MHz, $CDCl_3$) δ/ppm 1.323-1.346 (2H, m), 1.354-1.383 (2H, m), 1.649-1.738 (4H, s), 1.938-1.987 (2H, m), 7.284 (1H, s); ^{13}C {^{2}H}-NMR 145 MHz, $CDCl_3$) δ/ppm 18.88, 20.22, 25.82, 67.48, 76.73, 77.05, 77.37; GCMS (EI, 70 eV) calcd for $C_7H_{10}Cl_2$ (M+) 164, found 164 (s), (M+1) 165 (w), (M+2) 166 (m), (M+4) 168 (w).

4.0 CONCLUSIONS

This synthesis demonstrated phase-transfer catalysis methodology can produce the desired cyclopropanyl derivative.

The catalyst, triethylbenzylammonium chloride, along with sodium hydroxide, chloroform reacted to generate the carbene intermediate, which coupled with cyclohexene forming 7,7-dichloronorcarane. The latter reaction was a carbene/cyclohexene [1+2] cycloaddition. The reaction proceeded from aqueous and hydrophobic organic layers.

The crude product was extracted using dichloromethane with an initial yield of 8.73 g, dropping to 7.33 g after it was left for a week so any residual dichloromethane could evaporate. The percent yield of the crude product was 45%. Vacuum distilling the product at between 140 and 165 mm/Hg

passed the purified product at a boiling point of 143 °C. The isolate was colorless but slightly cloudy, with a mass of 5.69 g and a percent yield of 35%. The overall yield indicates this procedure could be potentially optimized.

No pronounced, broad peak was observed between 3200 and 3600 cm^{-1}, eliminating residual water as the cause of the cloudiness. Being water could have distilled over as a lower boiling water/7,7-dichloronorcane heteroazeotrope, it was a suspect contaminant. A very small signal on the ^{1}H NMR at a shift of 7.28 was observed where a chloroform or TEBA aromatic hydrogen signal would be expected. A very small peak at a little over 3000 cm^{-1} was also observed on the IR indicating a possible trace of unreacted cyclohexene present in the product. One possibility is vacuum grease was dissolved by the product during the distillation. No clear cause of the impurity was determined from the IR or NMRs.

5.0 REFERENCES

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6.0 SUPPLEMENTAL INFORMATION

6.1 Formulas

Number of moles

Equation S1

Eg

0.0987 mols = 8.1 g / 82.14 mols / g

Percent Yield

% yield = experimental yield / predicted yield * 100

Equation S2

Eg

45% yield = 7.33 / 16.60 * 100

Clausius-Clapeyron equation

$P_2/P_1 = e^{((1/T_1-1/T_2) *\Delta H_{vap}/R)}$

Equation S3

$P_2 = e^{((1/T_1-1/T_2) *\Delta H_{vap}/R)*P1}$

Trouton's Rule

$\Delta H_{vap}/ T_1 \approx 88J/(mol\ K)$

Equation S4

$\Delta H_{vap} \approx T_1 *88J/(mol\ K)$

Producing this rearranged equation:

$P_2 \approx P_1/e^{((1/T_2-1/T_1) *T_1*88/R)}$

Eg:

approximate boiling point at 1 atm – 204 °C (477.2 °K)

1 atm = 760 mm/Hg

Measured boiling point at reduced pressure – 143 °C (416.2 °K)

$P_2 \approx e^{((1/477.2-1/416.2) *477.2*88/R)*760}$

$P_2 \approx e^{((1/477.2-1/416.2) *477.2*88/8.314)*760}$

$P_2 \approx 0.212*760$

$P_2 \approx 161$ mm/Hg

6.2 Significant Figures

Significant figures used were the significant figures of the raw data plus one, for all calculation results.

6.3 Raw Data

Available on request.

6.4 Tables, Graphs, and Plots

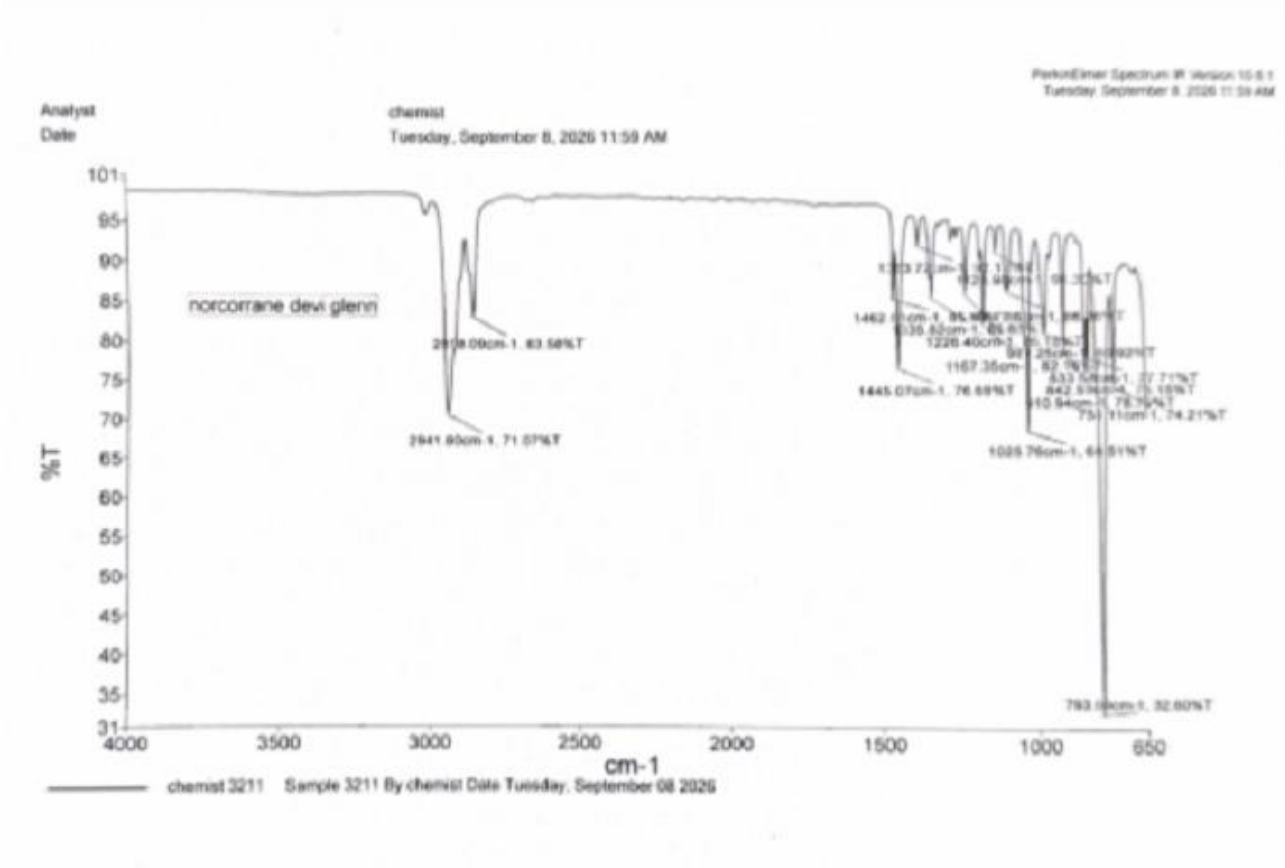


Figure S1 IR spectrum of distilled 7,7-dichloronorcane

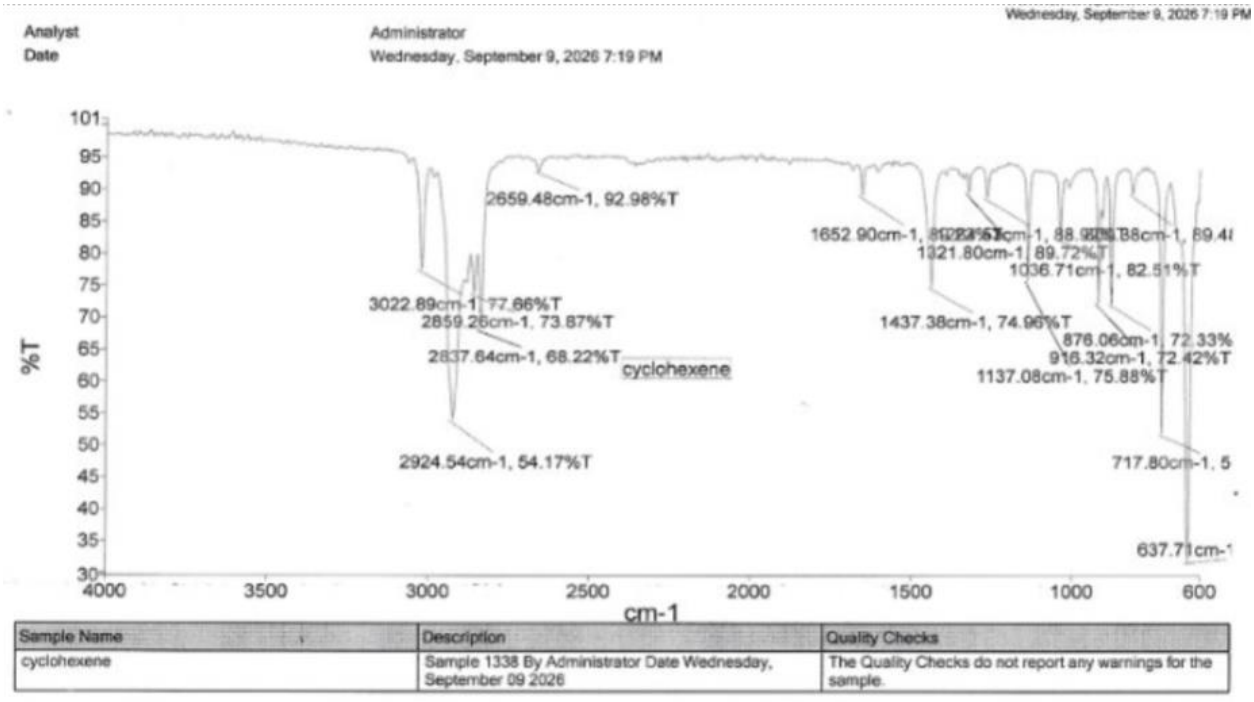


Figure S2 IR spectrum of cyclohexene

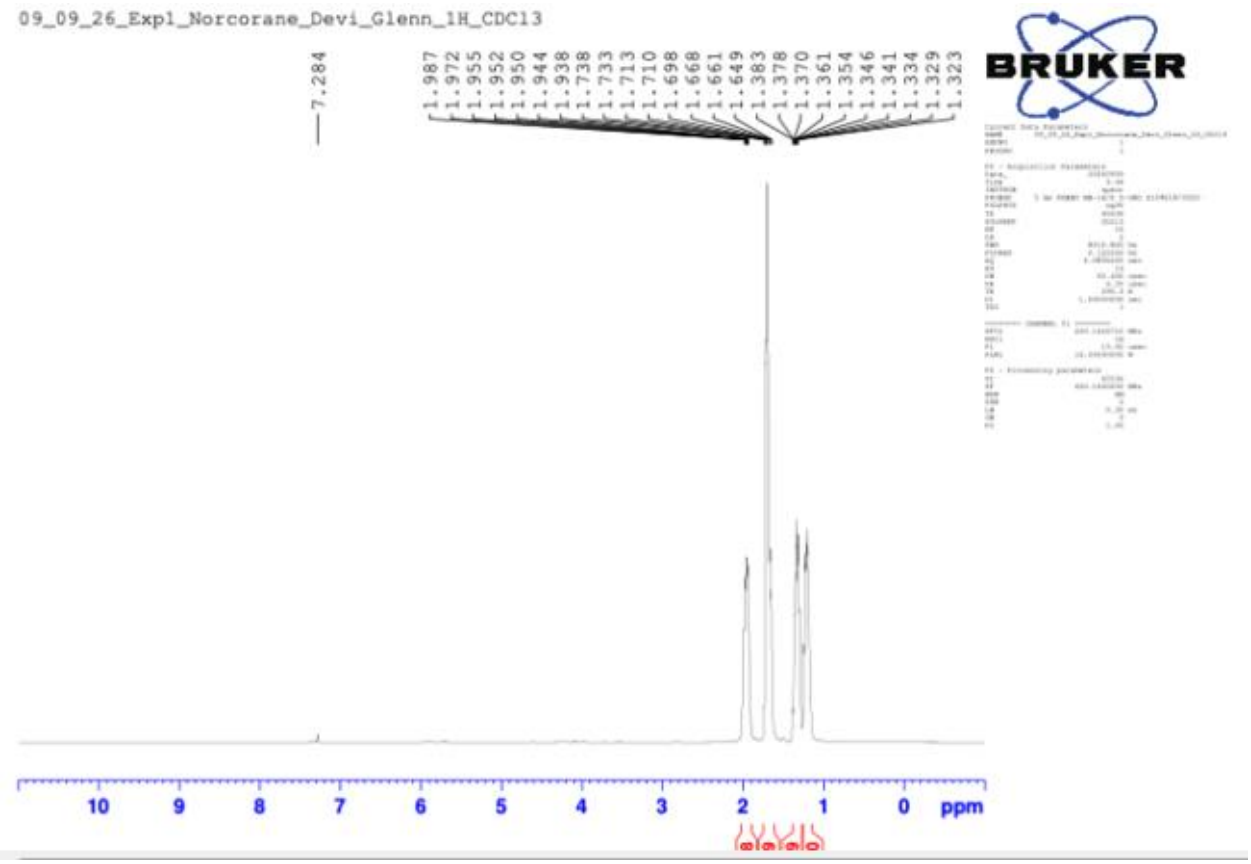


Figure S3a ^{1}H NMR of 7,7-dichloronorcarane

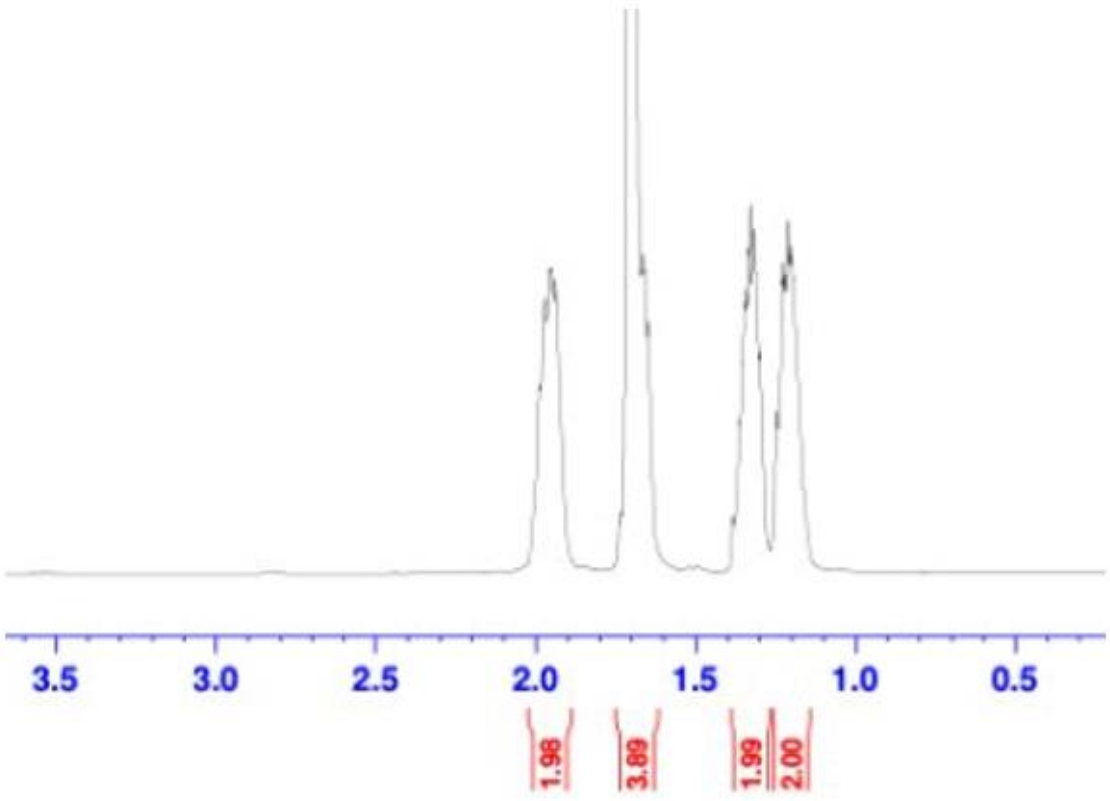


Figure S3b ^{1}H NMR of 7,7-dichloronorcarane Zoom

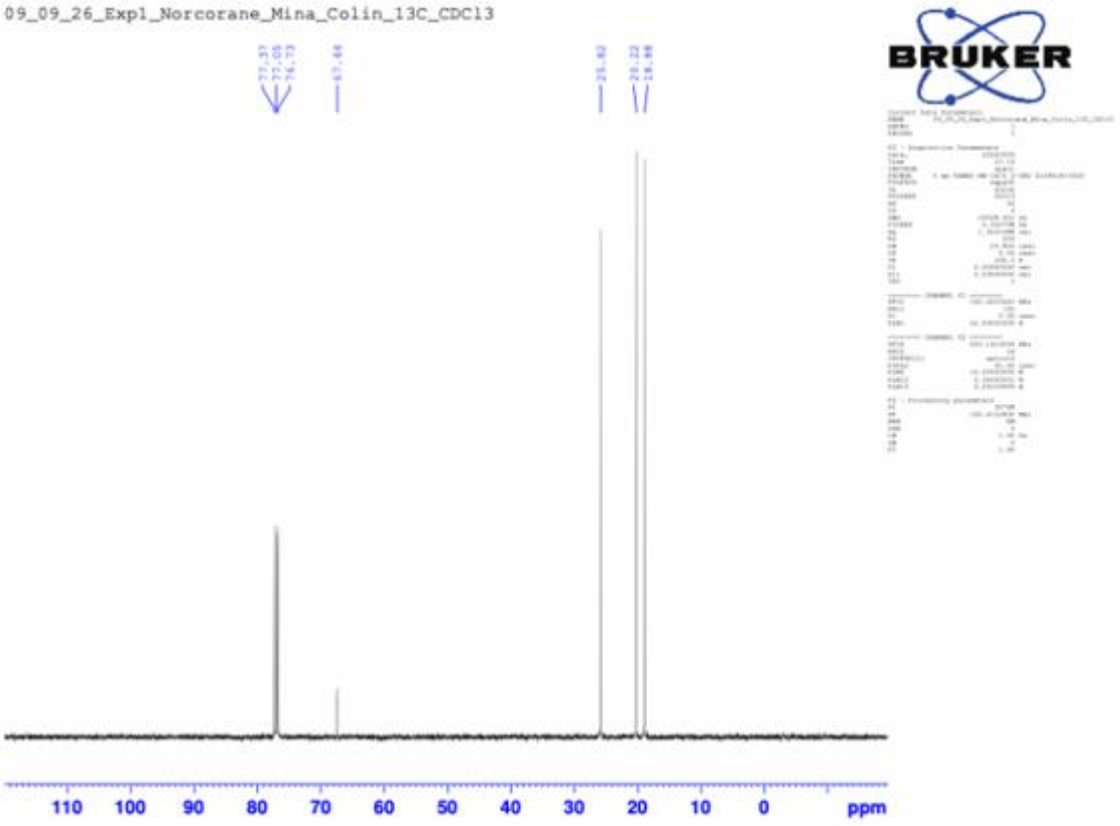


Figure S4 ^{13}C NMR of 7,7-dichloronorcarane - Sample

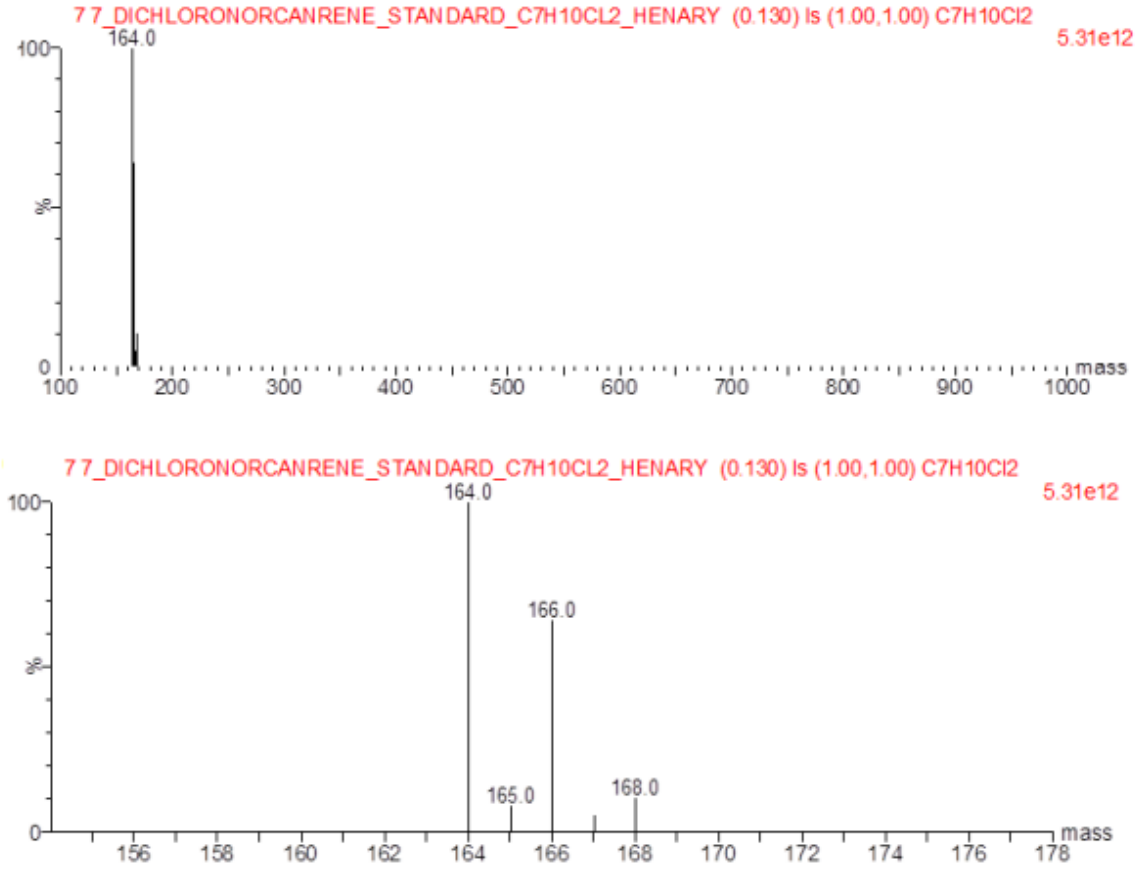
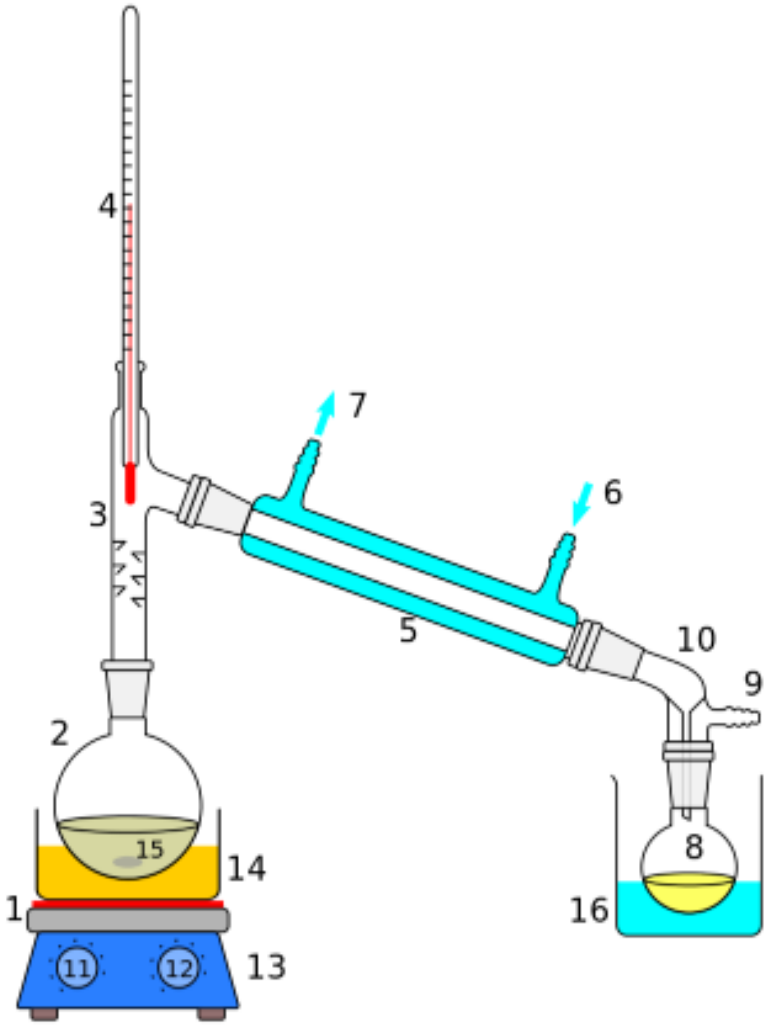


Figure S5 Mass spec from GC/MS



1. Heat source (usually a hot plate or Bunsen burner)
2. A round bottom flask that holds the mixture of liquids
3. Distillation head (where liquid travels as it evaporates)
4. Thermometer (to measure temperature and monitor boiling points)
5. Condenser (cools gas back down so it can return to liquid)
6. Water Cooling Hole (allows interaction with ambient temperature)
7. Water Cooling Hole (allows interaction with ambient temperature)
8. Rounded Flask (holds desired distillate)
9. Vacuum spout
10. Still Receiver (where distillate funnels into receiving flask)
11. Hot Plate controls
12. Hot Plate controls
13. Hot Plate controls
14. Heat Bath (ensures even heating without direct heat source contact)
15. Stir bar
16. Cooling bath (ensures even cooling)

Figure S6 Distillation diagram and labels from LabExchange

(https://www.labxchange.org/library/items/lb:LabXchange:84d2ffce:html:1)