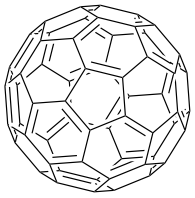
FIRST YEAR!

MISS FATTY ACID

A POLYATOMIC PAGEANT

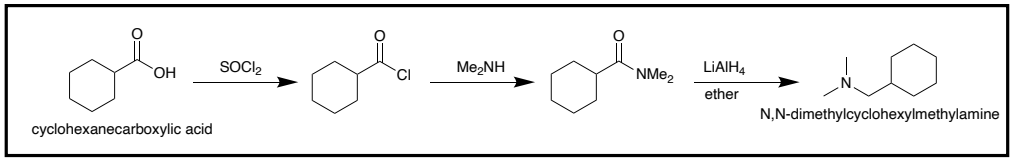


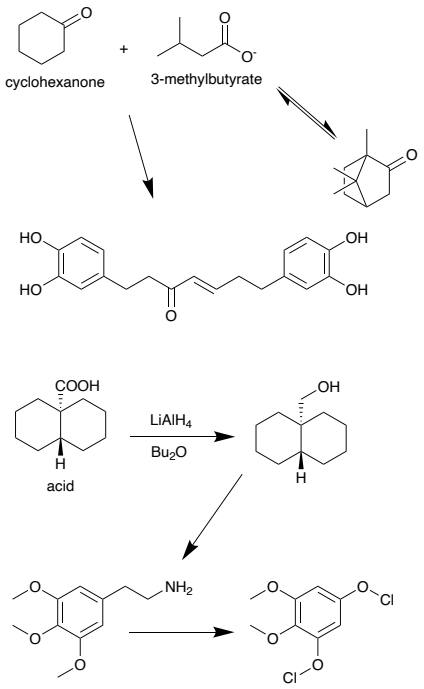
C69

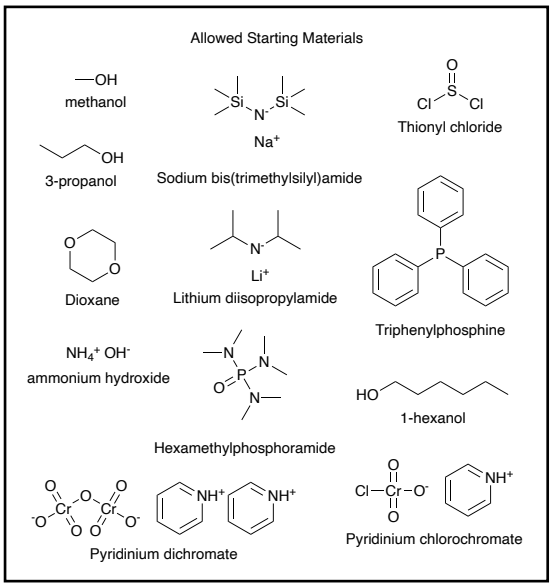
Competition Instruction Packet

See Website for Details Regarding Contest Rules and Submission Guidelines

PROTOCOL:







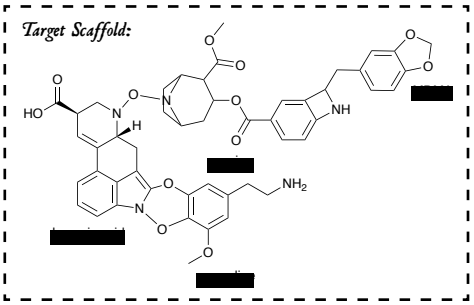
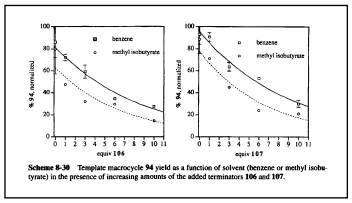
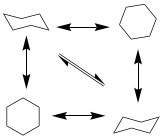


TABLE A3.4
RECRYSTALLIZING SOLVENTS

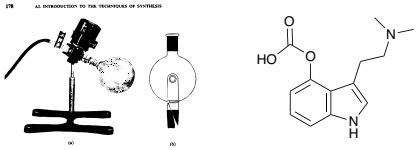
Solvent	B.P. (°C)	Dielectric constant	Water solubility (g/100 g)
Acetic acid	188	6.2	Misc.
Acetone	56.5	21	Misc.
Acetonitrile	82	38	Misc.
Benzene	80	2.3	0.07
t-Butyl alcohol	82	17	Misc.
Carbon tetrachloride	77	2.2	0.08
Chloroform	61	4.8	1.0
Cyclohexane	81	2.0	Sl. sol.
DMF	154	38	Misc.
Dioxane	101	2.2	Misc.
DMSO	189	45	Misc.
Ethanol	78	25	Misc.
Ethyl acetate	77	6.0	9
Ethyl ether	35	4.3	7.5
Ethylene chloride	83	10	0.83
Heptane	98	2.0	Insol.
Hexane	69	1.9	Insol.
Isopropyl alcohol	82	18	Misc.
Methanol	65	34	Misc.
Methylene chloride	40	9.1	2.0
Nitromethane	101	38	10
Pentane	36	2.0	0.03
Pyridine	115	12	Misc.
Water	100	80	—



Scheme 8-30 Template macrocycle **94** yield as a function of solvent (benzene or methyl isobutyrate) in the presence of increasing amounts of the added terminators **106** and **107**.



For preparation of N,N-Dimethylcydohexanecarboxamide: Clean 500-ml three-necked flask with reflux condenser, funnel, and magnetic stirrer. Charge with 36 g (0.25 mol) cydohexatrexaplexanide with dehydrated thonyl chloride (45 g, 0.375 mole) over 3.5 minutes, before boiling in oil bath for eleven hours.

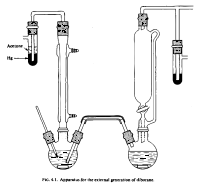


Note: Anal insertion of glassware not recommended

50 mL anhydrous benzene added before reheating to 95°. Cooled acid chloride immersed in ice bath; solution of 34 g (0.75 mole) anhydrous dimethylamine in 40 ml anhydrous benzene added to flask. Acid chloride solution stirred at room temperature over night. Water (50 ml) added to separate layers, aqueous phase extracted two 25 mL ether portions. Solvent removed (rotary evaporator), residue distilled under reduced pressure. The yield is about 16 g (88%) of A^-dimethylcydohexylmethylamin

NOTE: reaction vessel should be heated using steam bath, oil bath, or mantle. Lithium aluminum hydride (10g, 0.26 mole) dissolved in 200 ml dry n-butyl ether. Heat with stirring to 100°. 9.1 g (0.05 mol) trans-9-decalincarboxylic acid (See Supplement 7, Part C) in 100 ml dry n-butyl ether added drop-wise over 30 hrs. Stir, low heat 5 days, before cooling, slowly adding water to decompose hydride. Dilute HCl should be added to dissolve present salts, before separating and washing ether layer.

Fit 500-ml three-neck flask with thermometer, condensation tube, and gas dispersion apparatus. Condenser outlet should dip BELOW surface of mercury, which is covered in acetone layer; this will destroy excess diborane in the event of an emergency. 28.2g (0.30 mol) noroborene should be added to 100 ml THF, immediately immerse in reaction liquid via Tygon tubing connection. After equalizing pressure, solution of sodium borohydride (3.4g, 20% excess) added to the olefin-THF solution (20°) by the slow flow of nitrogen through the generation system. After one hour heat 70-85° to ensure complete transfer of the organoborane before unexpected peroxide oxidation occurs.



Caution: Nitrosyl chloride is an extremely corrosive gas, and all operations with it should be carried out in a hood.

Three catalysis routes are available, though only one will not result in the immediate, painful, and sudden death of the Chemist.

1) Via Phosphorous Pentoxide: 7-L round-bottom flask activated with 1-ethynylcydohexanol (40g, 0.32 mole), 250 ml dry benzene, and 10 g phosphorous pentoxide. Condenser added to flask and reagents massaged and steamed gently in a bath for eleven hours. Cooled solution washed, dried, and benzene removed (rotary evaporator); fractionalization produces product with bp 34-88°, about 25 g (61%).

2) Via Acidic Resin: Resin (Dowex-50, 200-400 mesh, or sulfonated polystyrene) prepared by suspension in sulfuric acid, followed by aggressive washing and air drying for forty-two months. 100 ml acetic acid, 10 ml urea, and 30 g resin refluxed seven minutes, turning brown to almost black. Product taken into ether, which is then immediately removed under vacuum to form desired distillate. Expect 85% yield.

3) Via Formic Acid: 65 g (0.5 mole) 1-ethynylcydohexanol and 400 ml formic acid (90%) heated under reflux until mild explosion occurs (preferably carried out in blast-resistant hood). To avoid structural isomerizations, two liters ice water used follow by pentane extraction. Wash with sodium hydroxide solution (10 %). Expected yield 32 g (49%).

Caution: The reaction and the subsequent solvent removal and product distillation steps must be carried out behind a safety screen, fully dothed with appropriate undergarments.

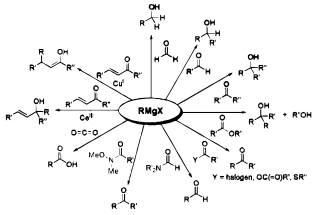


Diagram: Suggested Grignard Reagents

Bromine solution should be added at a rate of 1 drop per seven minutes over the course of 19 hours. followed by immediate addition of 50 ml dry carbon tetrachloride through funnel over one hour. After cooling, filter to remove mercuric bromide, wash with 5 % hydroxide, and fractionally distillate to give expected yield of 6 g (37%) 1-bromohexaniqpentanoic trilelol, bp 894-90°.

Final product must be appropriately sealed and submitted on or before the deadline via Express Overnight Shipping.
Incomplete or missing paperwork will result in immediate disqualification and a public shaming.

A COMPETITION PRESENTED BY...

THE CHEMISTRY LEAGUE OF
AMERICA, ISRAEL, RUSSIA, AND EUROPE

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