NOTEBOOK

The laboratory notebook should be well-organized and clear. Anyone looking at it should be able to determine exactly what was done and should be able to reproduce the experiment. Therefore, it is essential that the notebook be maintained during the course of the experiment, not afterward. All entries must be made using an ink pen. Any errors should be crossed out with a single line and the correction written above or next to the error. The notebook itself may be purchased in the stockroom and should be a bound volume with numbered and lined pages.

A proper notebook page should contain the following information:

1. The date.
2. The reaction being attempted, with appropriate references to the chemical literature.
3. A list of all reagents and solvents used, including molecular weights, sources (notebook page on which the compound was made or company from which it was purchased, including lot number and date), equivalents used, amounts used (mmol and weight or volume), and any relevant physical properties (density, boiling point, etc.).
4. A detailed procedure, including observations made during the reaction and a sketch of the experimental apparatus if it consists of more than simply a flask.
5. The yield of the reaction, including melting point if the product is a solid.
6. Copies of TLC plates, labeled with the eluent solvent system, developing method, identifiable spots, and R_f values.
7. A list of spectra taken, where each spectrum is labeled with the notebook page on which the corresponding experimental appears.
Compound | SW | t | N2O | AlOH | equ
-------|----|---|-----|------|----
M12 | 285.40 | 69.00 | 108.69 | 60.05 | 715.19
source | SPK-005 | Aldrich | Fisher | Fisher
eq | 2.0 eq | (2.2 Mism) | (1.0 M 1.5 M)
final | 1g | 0.48g | 17.2 mL | 3.4 mL | 1.10g (110%)
neutral | 7.0 and 6.8 mol
property: sold, sold | bp 138-140°C | bp 110-112°C

10:30. Placed 50 mL of 15% NaOH in 150 mL of 60% Schick flask at
stirrer under Ar.

10:35. Added 12.8 mL of 85% H2SO4 to Schick flask of 50 mL of
sodium hydroxide. Esterified with water to 75°C.

10:40. Added 0.188 M of NaOH, 50 mL of 85% H2SO4

11:10. The 9:1 H2SO4:NaOH solution was concentrated, and emerged (PMR)

12:30. Concentrated the mixture to dryness on vacuum line (collected residues
in 75 mls).

12:10. Took up residue in 100 mL of EtOAc, concentrated to 25 mL of aqueous

95% aqueous Na2CO3. Washed organic 2 x 40 mL

85% aqueous Na2CO3. Washed organic (3 x 20 mL)

13:30. Protocol residues by silica gel chromatography

13:45 hrs. (Rf 0.35, 0.38, 0.31)

Theoretical yield: 1.101 g (75%)

Actual: 1.048 g (94.9%)
8/8/92

Ref: White, E.J. JACS 1955 77 5028

Compound | MW | Value | D.C. | ALOH | Prod.
---------|----|-------|------|------|-------
         |    |       |      |      |       

Source: SPELTON, Aldrich, Baker, Fischer

eq. | 2.0 eq (0.1 M in DMF) | (1.0 M in 5mL)

mg | 0.485 | 17.20 | 3.10 | 1.018

netal | 7.41 and 6.8

property | solid, solid | bp 115-118°C | bp 116-118°C

- 10:30. Place 1.0 eq. in 50 mL of ethanol. Stirred under Ar.

- 10:35. Added 2.0 eq. A1203. To 10 mL of ethanol, cooled to 0°C.

- 10:40. Add 0.5 eq. of Na2CO3. Wash 3 times with 0°C.

- 10:45. The (7.1:1) fraction was then filtered. The filtrate was

- 11:30. Concentrated under vacuum to dryness on vacuum line (collected 37°C at 33 mm).

- 12:00. Take up residue in 100 mL of EtOAc, washed with 25 mL of 0.1 M HCl. The extracted aqueous

- 12:30. Washed combined organic 2.5 mL 0.1 M Na2CO3. Dried organic (Na2SO4, 2.0 mL).

- 12:40. Concentrated. Filtrated residue by silica gel chromatography.

- 12:50. Chromed derived as yellow oil.

Theoretical Yield: 1.1015 g (35 mmol)

Actual: 1.0450 g (34.9%)

NMR, FTIR, SPA T 0.71.1

FTIR: SPA T 0.71.1

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