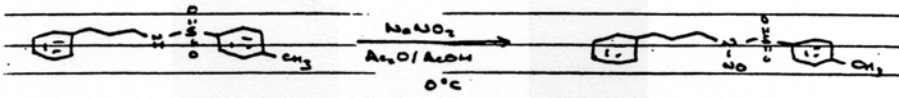
NOTEBOOK

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.

6/15/92



reference: White, E.H. JACS 1955 77 6008

Compound	SM	$NaNO_2$	Ac_2O	AcOH	prod
MW	289.40	69.00	102.09	60.05	318.29
source	SPA.I.070.1	Aldrich	Baker	Fisher	
eq		2.0 eq	(0.2 M in soln)	(1.0 M in soln)	
amount	1g	0.48 g	17.2 ml	3.4 ml	1.1018 g (theor)
mmol	3.4 mmol	6.8 mmol			
property	solid	solid	bp: 138-140°C	bp: 116-118°C	

- 10:30 · Placed 1.0015 g SM in flame-dried 50 ml Schlenk flask w/ stirbar under Ar
- 10:35 · Added 13 ml Ac_2O & 4 ml AcOH to Schlenk flask w/ SM via grad. cylinder & funnel. Cooled rxn mixture to 0°C in ice-H_2O bath.
- 10:40 · Added 0.4868 g $NaNO_2$, let rxn stir and warm to ~ 5°C.
- 12:10 · TLC (7:1 Hex:EA) shows SM consumed, one new spot (higher Rf than SM)
- 12:30 · Concentrated rxn mixture to dryness on vacuum line (collected solvents in 3rd trap).
- 12:50 · Took up residue in 100 ml Et_2O, transferred to 250 ml separatory funnel. Washed w/ 50 ml H_2O. Then extracted aqueous 2x 50 ml Et_2O. Washed combined organics 2x 40 ml 5% aqueous Na_2CO_3. Dried organics ($MgSO_4$), filtered, concentrated. Purified residue by silica gel chromatography (9:1 hex:EtOAc). Obtained desired as yellowish oil.

^1H NMR: SPA.I.071.1
FT-IR: SPA.I.071.1

Theoretical Yield: 1.1018 g (3.5 mmol)
Actual: 1.0460 g (94.9 %)

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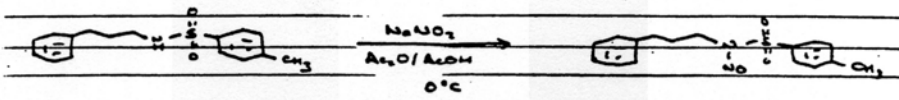

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