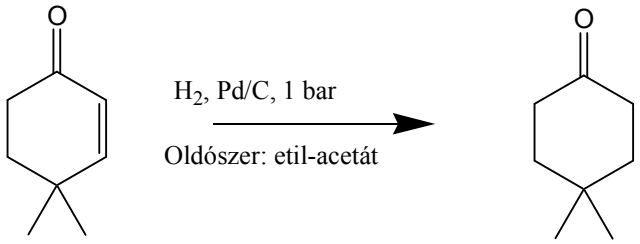
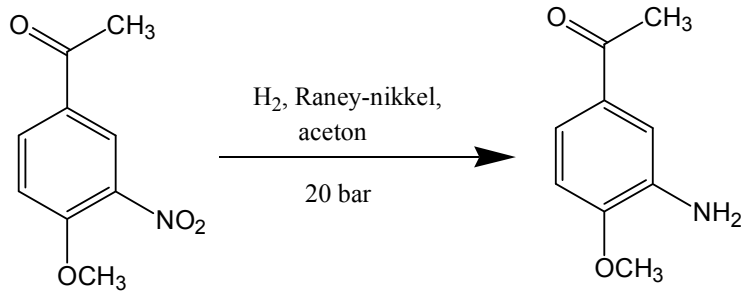
Etil-azodikarboxilát
Irodalom: Organic Syntheses, Coll. Vol. 3, p. 375.
Az első terméket, az etil-hidrazodikarboxilátot át kell kristályosítani.
A második terméket, az etil-azodikarboxilátot a hallgatók nem izolálják, számukra a reakció véget ér a számított mennyiségű klórgáz elnyeletésével és az oldat megszárításával.

Katalitikus redukciók

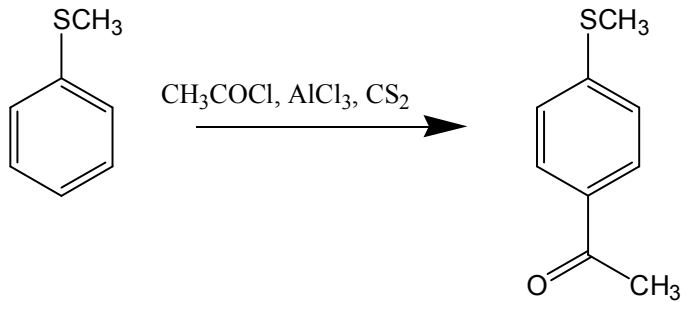
1. **Redukció légköri nyomáson Pd/C katalizátor jelenlétében hidrogén gázzal:**



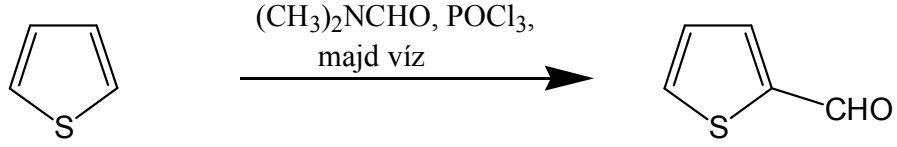
2. **Redukció 20 bar nyomáson Raney-nikkel katalizátor jelenlétében hidrogén gázzal, rozsdamentes acél autoklávban:**



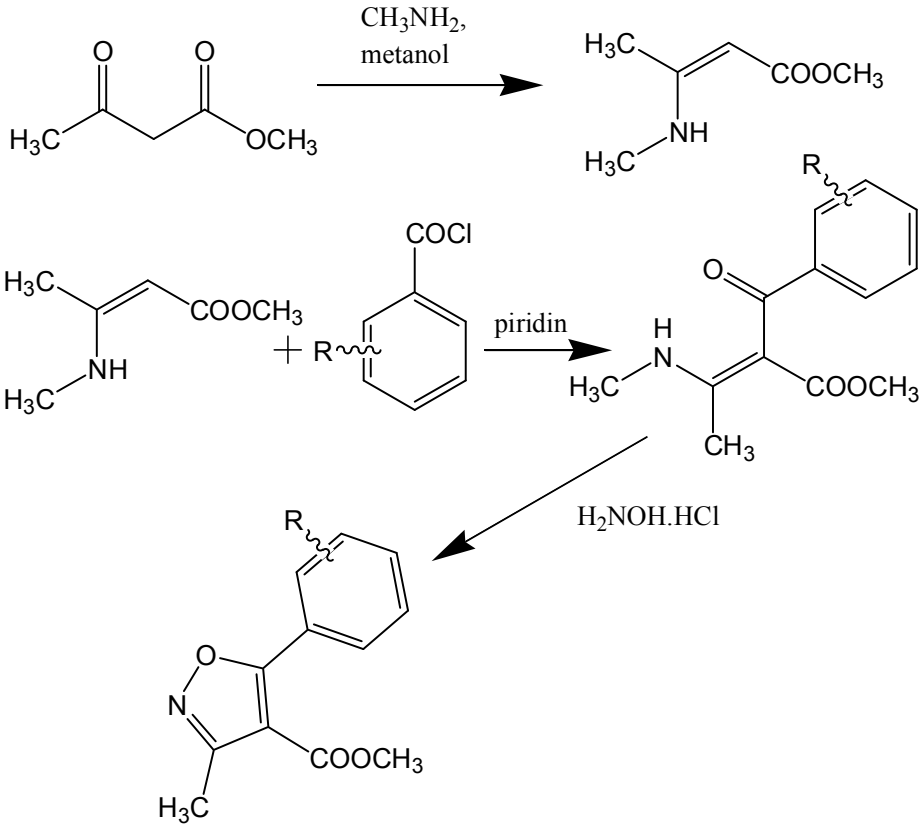
4-Metiltio-acetofenon (Friedel-Crafts acilezés). A szilárd nyerstermék desztillációja motorvákuumban, majd átkristályosítása.
Irodalom: J. Chem. Soc. 1952, 4175., 4-fluoro-3-methyl-acetophenone, azzal a különbséggel, hogy a reakcióelegyet 2 óra forralás után feldolgozzuk.



2-Formiltiofén (Vilsmeier-formilezés). A nyerstermék tisztítása frakcionált vákuumdesztillációval, motorvákuumban.
Irodalom: Vogel, Practical Organic Chemistry, 992. és 996-997. oldalak.



Heterociklusok: izoxazolvázas vegyületek szintézise.



Step 1: 3-Methylamino-but-2-enoic acid methyl ester

[00208] To a solution of methyl acetoacetate (29.4g, 253mmol) in Methanol (30mL) was added methylamine (33 wt% in EtOH; 48mL, 385mmol) dropwise at room temperature. The reaction was stirred for 1 hour, and then concentrated and dried to give the title compound as a white crystalline solid.

Step 2: 2-(4-Bromo-benzoyl)-3-oxo-butyric acid methyl ester

[00209] To 3-methylamino-but-2-enoic acid methyl ester (5.0g, 39.1mmol) in THF (70mL) was added pyridine (3.7mL, 47mmol) dropwise. The mixture was cooled to 0ºC, and 4-bromobenzoyl chloride (8.55g, 39.1mmol) in THF (30mL) was added dropwise. The reaction was stirred at room temperature overnight, and then water was added. The mixture was extracted with EtOAc, and the combined organic layers were washed with water, dried, filtered, and concentrated to give the title compound.

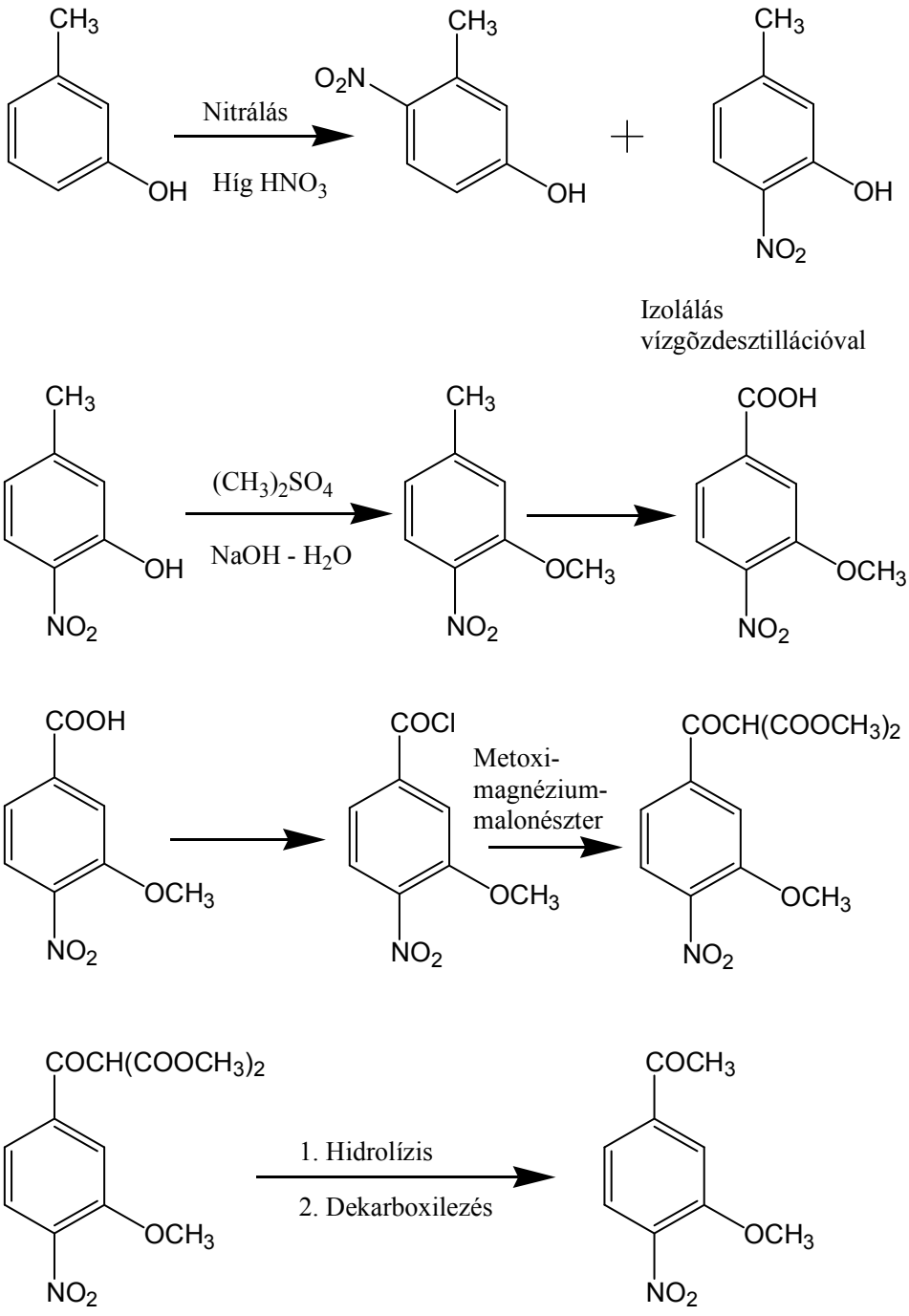
Step 3: 5-(4-Bromo-phenyl)-3-methyl-isoxazole-4-carboxylic acid methyl ester

[00210] To a mixture of 2-(4-bromo-benzoyl)-3-oxo-butyric acid methyl ester (11g, 39mmol) in acetic acid (50mL) was added hydroxylamine hydrochloride (2.66g, 39mmol), and the reaction was stirred at 115ºC for 1 hours. After cooling, saturated aqueous $NaHCO_3$ was added to the mixture to adjust to pH 8. The solution was extracted with EtOAc, and the combined organic layers were washed with brine, dried, filtered, and concentrated to give the title compound.

Step 4: 5-(4-Bromo-phenyl)-3-methyl-isoxazole-4-carboxylic acid

[00211] Lithium hydroxide (2g, 48mmol) was added to a solution of 5-(4-bromo-phenyl)-3-methyl-isoxazole-4-carboxylic acid methyl ester (39mmol) in methanol (50mL) and water (10mL), and the reaction was stirred at 60ºC for 1 hour. Acidic work-up gave the title compound.

3-Metoxi-4-nitro-acetofenon (több lépéses szintézis)



Irodalom (részben analóg receptekkel): nitrálás egyszerűen híg salétromsavval, a továbbiak

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Savkloridképzés a magyar Praktikum alapján tionil-kloriddal

Vogel: Practical Organic Chemistry, Experiment 5.96