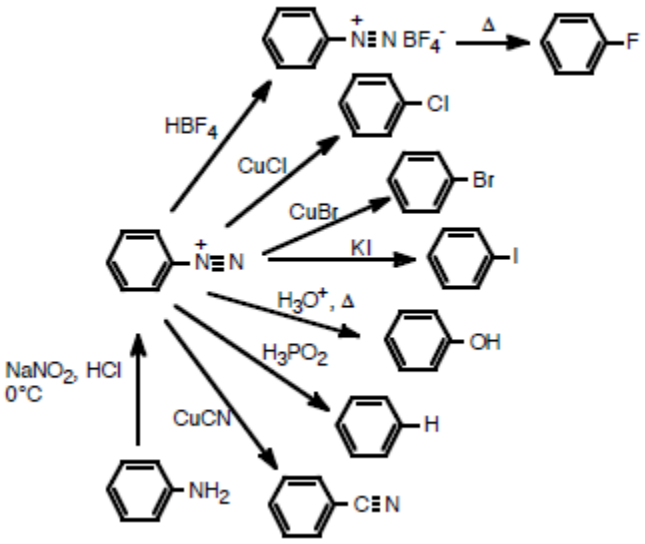
Ozonelabs Inc.
Organic chemistry syntheses

Synthesis of 2-Iodobenzoic Acid

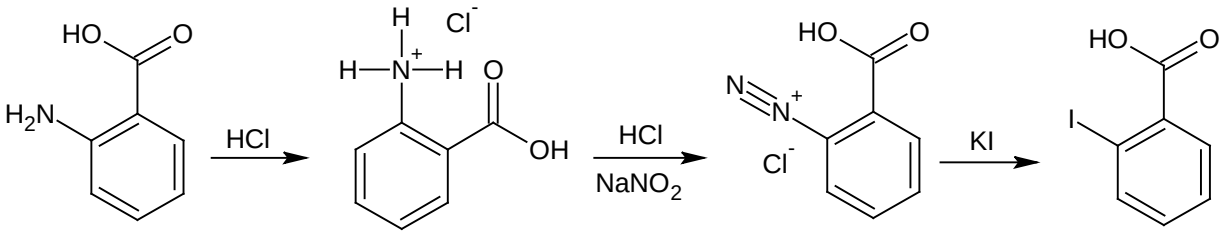
Introduction:

The Sandmeyer reaction is very well documented. In this case, the conversion of Anthranilic acid to 2-Iodobenzoic acid.

A brief summary of several possible Sandmeyer reactions is to the right.

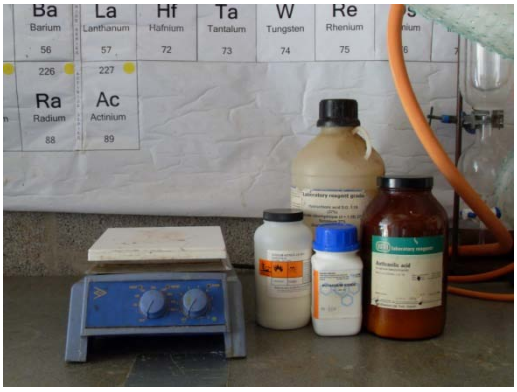


Reaction Scheme:



Reagents:

i) Anthranilic acid	BDH 99.5%
i) Sodium Nitrite	Breckland 99%
ii) Hydrochloric acid 38%	Fisher 99%
i) Potassium Iodide	Philip Harris 98%



Experimental:

In a 250mL Beaker, 6.8g of Anthranilic acid was dissolved in 50ml of water; made acidic by the addition of 12ml conc. HCl. The beaker was warmed on a hot plate until no solids remained. (Fig.1)

Fig. 1

The solution was then cooled in an ice bath. To the flask, 3.6g of Sodium nitrite in 25ml water was added slowly and the solution was stirred until everything dissolved. (Fig.2)

Fig. 2

After 5 minutes, a solution of 8.5 g of potassium iodide in 12 mL of water was added, a brown complex partially separated. The mixture was left to stand without cooling for 5 minutes
and then warmed on a hotplate to 45 °C with occasional swirling. When the reaction reached 40°C, a vigorous reaction ensued, liberating a small amount of Iodine vapour and a tan solid. After reacting for 10 minutes, the mixture is heated to 90°C
for 10 minutes on the hotplate and then cooled in ice. (Fig. 3)

Fig. 3

0.5g of Sodium thiosulphate was added to destroy any Iodine present and the product was vacuum filtered .

Fig. 4

The still-moist product was dissolved in 35mL of 95% Ethanol, and then diluted with 15mL of hot water. The solution was brought to the boiling point, filtered hot, diluted with 20mL of cold water, and allowed to stand. 2-Iodobenzoic acid separated in small brown needles (mp 163°C (lit. 164°C)) yield was 8.32g (71%).

Fig. 5

References

http://courses.chem.psu.edu/chem36/New%20Syn%2036%20pdf/Exp30.pdf