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Oxidative decarboxylation of α-amino acids to nitriles using *o*-iodoxybenzoic acid in aqueous ammonia *

Supplementary Information

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EXPERIMENTAL

General procedure for oxidative decarboxylation of α-amino acids

A solution of IBX (12.5 mmol) was prepared by adding it to aqueous ammonia (75 mmol) with stirring. To this solution after 5 min was added α-amino acid (5 mmol) in one portion and heating commenced. The reaction temperature was allowed raise to 75 ^{o}C and reaction continued until complete consumption of starting material as observed on TLC. After completion of reaction, reaction mixture was extracted with chloroform (2 x 15 mL). The organic layer was washed with water (2 x 10 mL), dried over anhydrous sodium sulfate and concentrated under reduced pressure to give crude product. Pure product was obtained after column chromatography (silica gel, # 60-120, eluent, ethyl acetate: hexane 5:95).

All the products obtained were known compounds

SPECTRAL DATA FOR NITRILES

Benzonitrile 2a

Colorless liquid, bp 190-191 ^{o}C, (lit.[1] bp 191 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 7.72-7.33 (m, 5H) ppm. IR (neat) ν_{max} = 2228 (CN) cm^{-1}.

2-Chlorobenzonitrile 2b

White solid, mp 41-44 ^{o}C (lit.[1] mp 43-45 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 7.75-7.55 (m, 2H), 7.39-7.35 (m, 2H) ppm. IR (KBr) ν_{max} = 2229 (CN) cm^{-1}.

4-Fluorobenzonitrile 2c

Solid, mp 35-37 ^{o}C (lit.[1] mp 36-39 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 7.99-7.64 (m, 2H), 7.46-7.01 (m, 2H) ppm. IR (KBr) ν_{max} = 2231 (CN) cm^{-1}.

Phenylacetonitrile 2d

Colorless liquid, bp 232-233 ^{o}C, (lit.[1] bp 233-234 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 7.32 (s, 5H), 3.68 (s, 2H) ppm. IR (neat) ν_{max} = 2250 (CN) cm^{-1}.

4-Methoxyphenylacetonitrile 2e

Colorless liquid, bp 285-287 ^{o}C, (lit.[1] bp 286-287 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 7.52-7.49(d, *J* = 8.4 Hz, 2H), 6.89-6.86(d, *J* = 8.4 Hz, 2H), 3.84(s, 3H), 3.75 (s, 2H) ppm. IR (neat) ν_{max} = 2250 (CN) cm^{-1}.

Pentanenitrile 2f

Colorless liquid, bp 139-140 ^{o}C, (lit.[1] bp 139-141 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 2.28-2.18 (t, 2H), 1.60-1.33(m, 4H), 1.08-0.99(t, 3H) ppm. IR (neat) ν_{max} = 2245 (CN) cm^{-1}.

2-Methylbutanenitrile 2g

Colorless liquid, bp 124-126 ^{o}C, (lit.[1] bp 125-126 ^{o}C). ^{1}H NMR (60 MHz, CCl_4): δ 2.68-2.24 (m, 1H), 1.86-1.50(m, 2H), 1.21-0.98 (m, 6H) ppm. IR (neat) ν_{max} = 2247 (CN) cm^{-1}.

3-Methylbutanenitrile 2h
Colorless liquid, bp 125-126 °C, (lit.[1] bp 125-126 °C). ^{1}H NMR (60 MHz, CCl_4): δ 2.27-2.23 (d, 2H), 2.20-21.99(m, 1H), 1.11-0.99 (d, 6H) ppm. IR (neat) ν_{max} = 2247 (CN) cm^{-1}.

Ethyl 3-cynopropanoate (3-cyanopropanoic acid **2i** was isolated as its ethyl ester)
Colorless liquid, bp 226-228 °C, (lit.[1] bp 228 °C). ^{1}H NMR (60 MHz, CCl_4): δ 4.21-4.10(t, *J* = 6.6 Hz, 2H), 2.76-273 (s, 4H), 1.23-1.03 (q, *J* = 6.6 Hz, 3H) ppm. IR (neat) ν_{max} = 2251 (CN), 1746 cm^{-1}.

3-Methylthiopropanenitrile 2j
Colorless liquid, bp 126-128 °C, (lit.[1] bp 127-130 °C). ^{1}H NMR (60 MHz, CCl_4): δ 2.84-2.57 (m, 4H), 2.33-2.19(m, 3H) ppm. IR (neat) ν_{max} = 2247 (CN) cm^{-1}.

Reference

1. *Dictionary of Organic Compounds*, Sixth Edition, Chapman and Hall electronic Publishing house, London.1996.