Supporting Information

Copper Catalyzed Electrophilic Amination of Diorganozinc Reagents

Ashley M. Berman and Jeffrey S. Johnson*

*Department of Chemistry, University of North Carolina at Chapel Hill,
Chapel Hill, North Carolina 27599-3290*

jsj@unc.edu

Experimental Section

Materials and Methods: General. Proton and carbon magnetic resonance spectra (^1H NMR and ^{13}C NMR) were recorded on a Bruker model Avance 400 (^1H NMR at 400 MHz and ^{13}C NMR at 100 MHz) spectrometer with solvent resonance as the internal standard (^1H NMR: CDCl_3 at 7.24 ppm and ^{13}C NMR: CDCl_3 at 77.0 ppm). ^1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, br s = broad singlet, d = doublet, br d = broad doublet, t = triplet, br t = broad triplet, q = quartet, m = multiplet), coupling constants (Hz), and integration. GLC analysis was performed on an Agilent 6890N Network GC System equipped with an HP-5 column (30 m x 0.316 mm, pressure = 10.0 psi, flow = 1.4 mL/min, detector = FID, 250°C) with helium gas as carrier. Analytical thin layer chromatography (TLC) was performed on Whatman 0.25 mm silica gel 60 plates. Visualization was accomplished with UV light and aqueous ceric ammonium molybdate solution followed by heating. Purification of the reaction products was carried out either by acid/base extractive work-up or flash chromatography using Sorbent Technologies silica gel 60 (32-63 μm). All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware with magnetic stirring. All transfers involving organometallic reagents were conducted in a dry box. Yield refers to isolated yield of analytically pure material. Yields are reported for a specific experiment and as a result may differ slightly from those found in the tables, which are averages of at least two experiments. Dichloromethane, diethyl ether, and tetrahydrofuran were dried by passage through a column of neutral alumina under nitrogen prior to use.¹ All diorganozinc reagents were prepared via transmetallation of the appropriate Grignard reagent, with the exception of bis-(2-pyridinyl)zinc which was prepared from the corresponding organolithium reagent, and diethylzinc which was purchased from Aldrich Chemical Company as a 1.0 M solution in hexanes. Unless otherwise noted, all other reagents were obtained from commercial sources and used without further purification. **CAUTION:** Dibenzoyl peroxide is potentially explosive and all reactions involving its use should be conducted with due care. While we have encountered no problems in the chemistry described herein, we advise the use of a blast and/or face shield.

General Procedure (A) for the copper catalyzed amination of diorganozinc reagents. An oven-dried round bottom flask equipped with magnetic stir bar was charged with the *N,N*-dialkyl *O*-benzoyl hydroxylamine derivative (0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0063 mmol), and anhydrous tetrahydrofuran (5.0 mL). A tetrahydrofuran solution of the diorganozinc reagent (2.1 mL, 0.26 M) was added and reaction mixture stirred at room temperature for 1 hour. The reaction mixture was diluted with diethyl ether (10 mL), the organic layer was washed with a saturated sodium bicarbonate solution (2 x 10 mL), and the aqueous layer was extracted with dichloromethane (2 x 10 mL). The organic layers were combined and the solvent was removed under reduced pressure. The resulting residue was taken up in diethyl ether (10 mL) and washed with a 1M hydrochloric acid solution (2 x 10 mL). The aqueous layer was basified with a 10% sodium hydroxide solution and extracted with dichloromethane (2 x 10 mL). The organic extracts were combined, dried over sodium sulfate, and the solvent was removed under reduced pressure, affording the desired product of ≥ 95 % purity as judged by ^1H NMR spectroscopy and GLC analysis (see Appendices A and B).

4-Phenylmorpholine. The title compound was prepared according to General Procedure A using 4-benzoyloxymorpholine (0.1031 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0028 g, 0.0056 mmol), and a tetrahydrofuran solution of diphenylzinc (2.1 mL, 0.26 M) to yield 4-phenylmorpholine (0.0797 g, 0.49 mmol, 98% yield) as a white solid. The ^1H NMR spectrum was consistent with that reported in the literature.²

4-(2-Methylphenyl)morpholine. The title compound was prepared according to General Procedure A using 4-benzoyloxymorpholine (0.1035 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0031 g, 0.0062 mmol), and a tetrahydrofuran solution of di-*o*-tolylzinc (2.1 mL, 0.26 M) to yield 4-(2-methylphenyl)morpholine (0.0858 g, 0.48 mmol, 97% yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.³

4-(4-Methoxyphenyl)morpholine. The title compound was prepared according to General Procedure

A using 4-benzoyloxymorpholine (0.1035 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0032 g, 0.0064 mmol), and a tetrahydrofuran solution of bis-(4-methoxyphenyl)zinc (2.1 mL, 0.26 M) to yield 4-(4-methoxyphenyl)morpholine (0.0911 g, 0.47 mmol, 94% yield) as a white solid. The ^1H NMR spectrum was consistent with that reported in the literature.²

4-Benzylmorpholine. The title compound was prepared according to General Procedure A using 4-benzoyloxymorpholine (0.1037 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0031 g, 0.0062 mmol), and a diethyl ether solution of dibenzylzinc (2.1 mL, 0.26 M) to yield 4-benzylmorpholine (0.0696 g, 0.39 mmol, 78% yield) as a clear colorless oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁴

4-Pyridin-2-yl-morpholine. The title compound was prepared according to General Procedure A using 4-benzoyloxymorpholine (0.1030 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0030 g, 0.0060 mmol), and a tetrahydrofuran solution of bis-(2-pyridinyl)zinc (2.1 mL, 0.26 M) to yield 4-pyridin-2-yl-morpholine (0.0586 g, 0.36 mmol, 71% yield) as a yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁵

1-Phenylpiperidine. The title compound was prepared according to General Procedure A using 1-benzoyloxypiperidine (0.1025 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0033 g, 0.0066 mmol), and a tetrahydrofuran solution of diphenylzinc (2.1 mL, 0.26 M) to yield 1-phenylpiperidine (0.0744 g, 0.46 mmol, 92% yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.²

1-(2-Methylphenyl)piperidine. The title compound was prepared according to General Procedure A using 1-benzoyloxypiperidine (0.1024 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene

complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0031 g, 0.0062 mmol), and a tetrahydrofuran solution of di-*o*-tolylzinc (2.1 mL, 0.26 M) to yield 1-(2-methylphenyl)piperidine (0.0839 g, 0.48 mmol, 96% yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁶

1-(4-Methoxyphenyl)piperidine. The title compound was prepared according to General Procedure A using 1-benzoyloxy-piperidine (0.1029 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0031 g, 0.0062 mmol), and a tetrahydrofuran solution of bis-(4-methoxyphenyl)zinc (2.1 mL, 0.26 M) to yield 1-(4-methoxyphenyl)piperidine (0.0938 g, 0.49 mmol, 98% yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁶

***N,N*-Diethylaniline.** The title compound was prepared according to General Procedure A using *N,N*-diethyl-*O*-benzoylhydroxylamine (0.0955 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0034 g, 0.0068 mmol), and a tetrahydrofuran solution of diphenylzinc (2.1 mL, 0.26 M) to yield *N,N*-diethylaniline (0.0574 g, 0.39 mmol, 77% yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁷

Diethyl-(2-methylphenyl)amine. The title compound was prepared according to General Procedure A using *N,N*-diethyl-*O*-benzoylhydroxylamine (0.0962 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0030 g, 0.0060 mmol), and a tetrahydrofuran solution of di-*o*-tolylzinc (2.1 mL, 0.26 M) to yield diethyl-(2-methylphenyl)amine (0.0650 g, 0.40 mmol, 80 % yield) as a pale yellow oil. The ^1H NMR spectrum was consistent with that reported in the literature.⁸

General Procedure (B) for the copper catalyzed amination of diorganozinc reagents. An oven-dried round bottom flask equipped with magnetic stir bar was charged with the *N,N*-dialkyl *O*-benzoyl hydroxylamine derivative (0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2 \cdot \text{C}_6\text{H}_6]$, 0.0063 mmol), and anhydrous tetrahydrofuran (5.0 mL). A tetrahydrofuran solution

of the diorganozinc reagent (2.1 mL, 0.26 M) was added and the reaction mixture was stirred at room temperature for 1 hour. The reaction mixture was diluted with diethyl ether (10 mL) and the organic layer was washed with a saturated sodium bicarbonate solution (2 x 10 mL), dried over magnesium sulfate, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography, eluting with the indicated solvent system, to afford the desired product of ≥ 95 % purity as judged by ^1H NMR spectroscopy and GLC analysis (see Appendix).

***N,N*-Dibenzylethylamine.** The title compound was prepared according to General Procedure **B** using *O*-benzoyl-*N,N*-dibenzylhydroxylamine (0.1585 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2] \cdot \text{C}_6\text{H}_6$, 0.0031 g, 0.0062 mmol), and a hexanes solution of diethylzinc (0.55 mL, 1.0 M) to yield *N,N*-dibenzylethylamine (0.1022 g, 0.46 mmol, 91% yield) as a pale yellow oil after flash chromatography with 10% ethyl acetate:hexanes. The ^1H NMR spectrum was consistent with that reported in the literature.⁹

***N,N*-Dibenzylisopropylamine.** The title compound was prepared according to General Procedure **B** using *O*-benzoyl-*N,N*-dibenzylhydroxylamine (0.1591 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2] \cdot \text{C}_6\text{H}_6$, 0.0029 g, 0.0058 mmol), and a tetrahydrofuran solution of diisopropylzinc (2.1 mL, 0.26 M) to yield *N,N*-dibenzylisopropylamine (0.0976 g, 0.41 mmol, 82% yield) as a clear colorless oil after flash chromatography with 2.5% ethyl acetate:hexanes. The ^1H NMR spectrum was consistent with that reported in the literature.¹⁰

***N,N*-Dibenzyl-*tert*-butylamine.** The title compound was prepared according to General Procedure **B** using *O*-benzoyl-*N,N*-dibenzylhydroxylamine (0.1589 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ($[\text{Cu}(\text{OTf})_2] \cdot \text{C}_6\text{H}_6$, 0.0031 g, 0.0062 mmol), and a tetrahydrofuran solution of di-*tert*-butylzinc (2.1 mL, 0.26 M) to yield *N,N*-dibenzyl-*tert*-butylamine (0.1250 g, 0.495 mmol, 99 % yield) as a white solid with no purification necessary. The ^1H NMR spectrum was consistent with that reported in the literature.¹¹

***N,N*-Dibenzylaniline.** The title compound was prepared according to General Procedure **B** using *O*-benzoyl-*N,N*-dibenzylhydroxylamine (0.1585 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ([CuOTf]₂·C₆H₆, 0.0033 g, 0.0066 mmol), and a tetrahydrofuran solution of diphenylzinc (2.1 mL, 0.26 M) to yield *N,N*-dibenzylaniline (0.1286 g, 0.47mmol, 94 % yield) as a white solid after flash chromatography with 1% ethyl acetate:hexanes. The ¹H NMR spectrum was consistent with that reported in the literature.¹²

Tribenzylamine. The title compound was prepared according to General Procedure **B** using *O*-benzoyl-*N,N*-dibenzylhydroxylamine (0.1582 g, 0.50 mmol), copper(I) trifluoromethanesulfonate benzene complex ([CuOTf]₂·C₆H₆, 0.0031 g, 0.0062 mmol), and a diethyl ether solution of dibenzylzinc (2.1 mL, 0.26 M) to yield tribenzylamine (0.1481 g, 0.46mmol, 92 % yield) as a white solid after flash chromatography with 5% ethyl acetate:hexanes. The ¹H NMR spectrum was consistent with that reported in the literature.¹³

General Procedure (C) for the preparation of *N,N*-dialkyl *O*-hydroxylamine derivatives. A modified procedure of that described by Ganem and Biloski was followed.¹⁴ An oven-dried round bottom flask equipped with magnetic stir bar and reflux condenser was charged with disodium hydrogen phosphate (7.1 g, 50 mmol), dibenzoyl peroxide (2.66 g, 11 mmol, **CAUTION:** Dibenzoyl peroxide is potentially explosive and all reactions involving its use should be conducted with due care; we advise the use of a blast and/or face shield), and anhydrous diethyl ether (100 mL). To this well stirred suspension was added the dialkyl amine (11 mmol) and the reaction mixture was stirred at reflux for 12 hours. The reaction mixture was allowed to cool to room temperature, the solvent was removed under reduced pressure, and the resulting residue was taken up in dichloromethane (50 mL). The organic layer was washed with saturated sodium carbonate solution (2 x 50 mL), saturated sodium chloride solution (1 x 50 mL), dried over sodium sulfate, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography, eluting with the indicated solvent system, to afford the desired product of ≥95 % purity as judged by ¹H NMR and ¹³C NMR spectroscopy (see Appendix A). The product was stored at -35°C under anhydrous conditions. For additional analytical data (melting points of all derivatives) see Ganem and Biloski.¹⁴

4-Benzoyloxy-morpholine. The title compound was prepared according to General Procedure C using morpholine (0.96 g, 0.96 mL, 11 mmol) to yield 4-benzoyloxy-morpholine (1.11 g, 5.4 mmol, 49% yield) as a white solid after flash chromatography with 40% ethyl acetate:hexanes. Analytical data for the title compound: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.00 – 7.98 (m, 2H), 7.58 – 7.53 (m, 1H), 7.45 – 7.41 (m, 2H), 3.96 (br d, J = 10.7 Hz, 2H), 3.85 (br t, J = 11.2 Hz, 2H), 3.43 (br d, 9.3 Hz, 2H), 3.03 (br t, 9.4 Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 164.6, 133.1, 129.4, 129.2, 128.4, 65.8, 57.0.

1-Benzoyloxy-piperidine. The title compound was prepared according to General Procedure C using piperidine (0.94 g, 1.1 mL, 11 mmol) to yield 1-benzoyloxy-piperidine (1.35 g, 6.6 mmol, 60% yield) as a white solid after flash chromatography with 15% ethyl acetate:hexanes. Analytical data for the title compound: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.00 – 7.98 (m, 2H), 7.55 – 7.51 (m, 1H), 7.43 – 7.38 (m, 2H), 3.49 (br s, 2H), 2.76 (br s, 2H), 1.84 – 1.78 (m, 4H), 1.65 (br s, 1H), 1.28 – 1.26 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 164.7, 132.8, 129.7, 129.4, 128.3, 57.5, 25.0, 23.3.

O-benzoyl-N,N-dibenzylhydroxylamine. The title compound was prepared according to General Procedure C using dibenzylamine (2.17 g, 2.1 mL, 11 mmol) to yield O-benzoyl-N,N-dibenzylhydroxylamine (2.37 g, 7.43 mmol, 68% yield) as a white solid after flash chromatography with 1% ethyl acetate:hexanes. Analytical data for the title compound: $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.82 – 7.80 (m, 2H), 7.50 – 7.46 (m, 1H), 7.44 – 7.42 (m, 4H), 7.36 – 7.23 (m, 8H), 4.19 (s, 4H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ 164.9, 136.0, 132.8, 129.5, 129.4, 129.3, 128.3, 128.28, 127.6, 62.1.

Procedure for the preparation of N,N-diethyl-O-benzoylhydroxylamine. An oven-dried round bottom flask equipped with magnetic stir bar and addition funnel was charged with N,N-diethylhydroxylamine (0.89 g, 1.0 mL, 10 mmol), freshly distilled triethylamine (1.0 g, 1.4 mL, 10 mmol), and anhydrous dichloromethane (10 mL). The addition funnel was charged with benzoyl chloride (1.4 g, 1.2 mL, 10 mmol) and anhydrous dichloromethane (5 mL). The benzoyl chloride

solution was added drop wise over 10 minutes and reaction mixture stirred at room temperature an additional 30 minutes. The reaction mixture was diluted with water (25 mL) and the organic layer was separated, washed with water (1 x 25mL), dried over sodium sulfate, and the solvent was removed under reduced pressure. The crude product was purified by flash chromatography, eluting with 15 % ethyl acetate:hexanes, to afford *N,N*-diethyl-*O*-benzoylhydroxylamine (1.89 g, 9.8 mmol, 98 % yield) as a pale yellow oil. The product was stored at -35°C under anhydrous conditions. Analytical data for the title compound: ¹H NMR (400 MHz, CDCl₃) δ 8.07 – 8.04 (m, 2H), 7.57 – 7.53 (m, 1H), 7.44 – 7.40 (m, 2H), 2.72 (q, *J* = 7.6 Hz, 4H), 0.73 (t, *J* = 7.6 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 165.8, 132.9, 129.4, 129.3, 128.3, 53.4, 11.8. For additional analytical data (IR spectrum), see Grinberg and Bittner.¹⁵

General Procedure for the preparation of diorganozinc reagents. An oven-dried round bottom flask equipped with magnetic stir bar was charged with anhydrous zinc chloride (0.0750 g, 0.55 mmol) and anhydrous tetrahydrofuran (1 mL). To this stirred solution was cautiously added a tetrahydrofuran or diethyl ether solution of the Grignard or organolithium reagent (1.1 mL, 1.0 M) and the reaction mixture was stirred at room temperature for 20 minutes. The diorganozinc reagent was used as is (0.26 M) without titrametric determination of concentration.

Conditions Evaluated for the Transition-Metal Catalyzed Amination of Organometallic Reagents

$$R_2N-X + Ph-M' \xrightarrow[\text{(Additive)}]{M (20 \text{ mol } \%) / \text{Ligand}} R_2N-M'$$

		ligand							
		no ligand	Imes-HCl	P(OPh) ₃	PPh ₃	binap	phen	blpy	
transition-metal salt	Pd(dba) ₂	○	○	○	○	○	○	○	PhB(OH) ₂ / Additive ^b
	Ni(cod) ₂	○	○	○	○	○	○	○	
	CuI	○	○	○	○	○	○	○	
	Pd(dba) ₂	○	○	○	○	○	○	○	PhSnBu ₃ / Additive ^b
	Ni(cod) ₂	○	○	○	○	○	○	○	
	CuI	○	○	○	○	○	○	○	
	Pd(dba) ₂	○	○	○	○	○	○	○	PhZnCl /
	Ni(cod) ₂	○	○	○	○	●	○	●	
	CuCN	●	○	●	○	●	●	●	
	Fe(acac) ₃	●	○	○	○	○	○	○	
	Pd(dba) ₂	○	○	○	○	○	○	○	Ph ₃ In /
	Ni(cod) ₂	○	○	○	○	○	○	●	
	[Cu(OTf)] ₂ · C ₆ H ₆	●	○	○	○	○	○	○	
	Fe(acac) ₃	●	○	○	○	○	○	○	Ph ₂ Zn /
	[Cu(OTf)] ₂ · C ₆ H ₆	●	○	○	○	○	○	○	
	Fe(acac) ₃	○	○	○	○	○	○	○	

% yield^a

○ 0-10 %
● 10-20 %
● 20-40 %
● 70-100 %

a) Yield determined by GLC versus undecane as an internal standard and is based on the starting R₂N-X. b) Additives tested: Na₂CO₃, K₂CO₃, Cs₂CO₃, K₃PO₄, CsF, KF, LiCl

References

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c1ccccc1N2CCCCO2

Et₂N-Ph

Bn₂N-OBz

Appendix B – GLC Traces

* RUN # 668 JUN 15, 1901 19:47:04

START

RUN# 668 JUN 15, 1901 19:47:04

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.922	26229472	SPB	.051	95.69267
9.401	1180637	PB	.101	4.30731

TOTAL AREA=2.7410E+07
MUL FACTOR=1.0000E+00

Oven: 130°C
Run Time: 20 min

RUN # 670 JUN 19, 1901 16:08:28

START

RUN# 670 JUN 19, 1901 16:08:28

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.945	41148192	SPB	.080	85.60086
9.594	6921645	PB	.082	14.39915

TOTAL AREA=4.8070E+07
MUL FACTOR=1.0000E+00

Oven: 130°C
Run Time: 20 min

RUN # 716 SEP 11, 1901 14:10:07

RUN# 716 SEP 11, 1901 14:10:07

AREAX

RT	AREA	TYPE	WIDTH	AREAX
1.955	24347376	SPB	.047	84.92397
13.980	43865	BB	.093	.15300
21.615	4278378	PB	.106	14.92304

TOTAL AREA=2.8670E+07

MUL FACTOR=1.0000E+00

Oven: 120°C to 160°C (1°C/min)

Run Time: 41 min

RUN # 717 SEP 12, 1901 23:08:54

RUN# 717 SEP 12, 1901 23:08:54

AREAX

RT	AREA	TYPE	WIDTH	AREAX
1.950	26354176	SPB	.050	73.11309
2.433	52505	BB	.026	.14566
2.635	131431	PB	.030	.36462
7.057	9507667	SBB	.063	26.37665

TOTAL AREA=3.6046E+07

MUL FACTOR=1.0000E+00

Oven: 140°C

Run Time: 20 min

* RUN # 722 SEP 14, 1901 13:23:18
START

RUN# 722 SEP 14, 1901 13:23:18

RT	AREA	TYPE	WIDTH	AREA%
1.942	39814624	SBB	.077	91.24874
2.665	159990	BB	.029	.36667
3.215	48185	BB	.071	.11043
8.233	3610266	SBB	.096	8.27416

TOTAL AREA=4.3633E+07
MUL FACTOR=1.0000E+00

Oven: 140°C
Run Time: 20 min

*
* RUN # 667 JUN 15, 1901 19:27:27
START

RUN# 667 JUN 15, 1901 19:27:27

RT	AREA	TYPE	WIDTH	AREA%
1.972	41056000	SPB	.080	89.90253
8.732	4611245	PB	.075	10.09749

TOTAL AREA=4.5667E+07
MUL FACTOR=1.0000E+00

Oven: 130°
Run Time: 20 min

* RUN # 671 JUN 21, 1901 15:16:59
START

RUN# 671 JUN 21, 1901 15:16:59

RT	AREA	TYPE	WIDTH	AREA%
1.973	40936800	SBB	.080	84.42464
8.633	7552365	PB	.072	15.57537

TOTAL AREA=4.8489E+07
MUL FACTOR=1.0000E+00

Oven: 130°C
Run Time: 20 min

RUN # 723 SEP 19, 1901 23:44:29
START

RUN# 723 SEP 19, 1901 23:44:29

RT	AREA	TYPE	WIDTH	AREA%
1.978	38217600	SPB	.072	73.14051
2.305	25532	BB	.024	.04886
10.312	140366	BB	.083	.26863
16.233	13868824	PB	.126	26.54204

TOTAL AREA=5.2252E+07
MUL FACTOR=1.0000E+00

Oven: 140°C
Run Time: 20 min

Et₂N-Ph

* RUN # 669 JUN 17, 1901 23:17:48
START

RUN# 669 JUN 17, 1901 23:17:48

RT	AREA	TYPE	WIDTH	AREA%
1.971	41494208	SBB	.081	89.29795
4.468	4972938	SBB	.052	10.70205

TOTAL AREA=4.6467E+07
MUL FACTOR=1.0000E+00

Oven: 130°C
Run Time: 20 min

* RUN # 672 JUN 23, 1901 22:30:28
START

RUN# 672 JUN 23, 1901 22:30:28

RT	AREA	TYPE	WIDTH	AREA%
1.970	40962080	SBB	.080	81.97299
3.917	8939610	SPB	.039	17.88988
7.610	28477	BU	.150	.05699
9.216	40067	BP	.071	.08018

TOTAL AREA=4.9970E+07
MUL FACTOR=1.0000E+00

Oven: 130°C
Run Time: 20 min

Bn₂N-Et

* RUN # 700 AUG 11, 1901 20:35:05

START

STOP

RUN# 700 AUG 11, 1901 20:35:05

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.921	26244784	SPB	.051	64.16090
2.533	60009	BB	.026	.14670
3.376	17329	PB	.056	.04236
8.659	74971	PB	.153	.18328
20.309	14507552	PB	.090	35.46677

TOTAL AREA=4.0905E+07

MUL FACTOR=1.0000E+00

Oven: 160°C

Run Time: 30 min

Bn₂N-ⁱPr

* RUN # 697 AUG 11, 1901 00:59:49

START

STOP

RUN# 697 AUG 11, 1901 00:59:49

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.921	26331056	SPB	.051	68.11946
2.536	19460	BB	.025	.05034
3.591	10488	PB	.039	.02713
22.835	12293616	PB	.101	31.80389

TOTAL AREA=3.8655E+07

MUL FACTOR=1.0000E+00

Oven: 160°C

Run Time: 30 min

Bn₂N-^tBu

* RUN # 698 AUG 11, 1901 18:08:37
START

RUN# 698 AUG 11, 1901 18:08:37

RT	AREA	TYPE	WIDTH	AREAX
1.930	26174496	SPB	.051	90.13674
2.535	14029	BB	.024	.04831
20.545	90407	BB	.098	.31133
24.964	2759722	PB	.114	9.50362

TOTAL AREA=2.9039E+07
MUL FACTOR=1.0000E+00

Oven: 160°C
Run Time: 30 min

Bn₂N-Ph

RUN # 720 SEP 13, 1901 13:40:21
START

RUN# 720 SEP 13, 1901 13:40:21

RT	AREA	TYPE	WIDTH	AREAX
2.055	29317024	SBB	.057	89.58506
7.486	3408422	BB	.421	10.41495

TOTAL AREA=3.2726E+07
MUL FACTOR=1.0000E+00

Oven: 180°C
Run Time: 20 min

Bn₃N

* RUN # 719 SEP 13, 1981 01:22:36
START

RUN# 719 SEP 13, 1981 01:22:36

RT	AREA	TYPE	WIDTH	AREA%
2.049	29355952	SPB	.056	98.47738
4.390	453086	PB	.038	1.52260

TOTAL AREA=2.9810E+07
MUL FACTOR=1.0000E+00

Oven: 180°C
Run Time: 20 min