Supporting Information

Tandem Formation and [2,3] Rearrangement of Methylene Ammonium Ylides Derived from Amines and the Simmons-Smith Reagent

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I Experimental Procedures and spectral data

General procedures

All air and water sensitive reactions were carried out in oven dried glassware under an argon atmosphere. Solvents were dried by standard methods.¹ NMR spectra were recorded on Jeol 270 MHz or Jeol 400 MHz spectrometers. Chemical shifts are given in ppm. CI mass spectra were obtained using a VG Platform machine. IR spectra were obtained on a Perkin Elmer Spectrum One FT-IR spectrometer. Flash chromatography was performed on silica gel (Merck Kiesegel 60). Melting points were determined with a Khofler hot stage apparatus and were not corrected.

General Procedures for 8a-d

N-methylalkylamine (15mmol) was mixed with NaHCO₃ (30mmol) in DMF (15mL). To this mixture was added the allylic bromide (16.5 mmol in 5mL DMF) over 3 hours at 70 °C. The reactants were stirred for a further 2 hours and quenched with water (100mL). The mixture was extracted with petrol (2 x 30mL), and then the combined organic phases were dried over Na₂SO₄ and concentrated under reduced pressure. The crude products were purified by distillation under vacuum.

N-Methyl-*N*-phenethyl-allyl amine 8a

The product was obtained as a pale yellow oil, yield: 50%, b.p.: 104-106 °C/12 mmHg, $\nu_{\text{max}}/\text{cm}^{-1}$ (neat) 3064, 3027, 2946, 1643, 1604, 1453. ¹H NMR (270 MHz, CDCl₃) 7.17-7.31 (5H, m, ArH), 5.88 (1H, ddt, J=17.2, 10.2, 6.6 Hz, CH=CH₂), 5.19 (1H, ddd, J=17.2, 3.3, 1.5 Hz, CH=CHH(*cis*)), 5.13 (1H, ddt, J=10.2, 3.3, 1.2 Hz, CH=CHH(*trans*)), 3.07, (2H, d, J=6.6 Hz, NCH₂CH=), 2.84-2.74 (2H, m, PhCH₂CH₂N), 2.65-2.57 (2H, m, PhCH₂CH₂N), 2.28 (3H, s, NCH₃). ¹³C NMR (68 MHz, CDCl₃) 140.5, 135.6, 128.7, 128.4, 126.0, 117.6, 60.9, 59.1, 42.1, 34.0. m/z (CI) 176 (M+1, 28), 147 (100), 105 (45). HRMS found: 176.1436, C₁₂H₁₈N required 176.1439.

N-Methyl-*N*-(3-methyl-but-2-enyl)-phenethyl-amine 8b

The product was obtained as a colorless oil, yield: 84%, b.p.: 128-130 °C/8 mmHg, $\nu_{\text{max}}/\text{cm}^{-1}$ (neat) 3060, 3027, 2929, 2786, 1604, 1452. ¹H NMR (270 MHz, CDCl₃) 7.19-7.30 (5H, m, ArH), 5.27 (1H, t, J=7.0 Hz, CH=C), 3.02 (2H, d, J=7.0 Hz, NCH₂CH=), 2.83-2.77 (2H, m, PhCH₂CH₂N), 2.64-2.58 (2H, m, PhCH₂CH₂N), 2.29 (3H, s, NCH₃), 1.74 (3H, s, CH=CCH₃(*trans*)), 1.65 (3H, s, CH=CCH₃(*cis*)). ¹³C NMR (101 MHz, CDCl₃) 140.7,

135.0, 128.7, 128.4, 126.0, 121.5, 59.3, 52.2, 42.1, 34.2, 26.0, 18.1. m/z (CI) 204 (M+1, 46), 148 (35), 112 (100). HRMS found: 204.1755, C₁₄H₂₂N required 204.1752.

***N*-Methyl-*N*-phenethyl-(3-phenyl-allyl)-amine 8c**

The product was purified by flash chromatography (20% ethyl acetate in petrol, R_f: 0.32) and obtained as a pale yellow oil. yield: 50%. ¹H NMR² (400 MHz CDCl₃) 7.33 (10H, m, ArH), 6.51 (1H, d, J=15.8 Hz, PhCH=), 6.28 (1H, dt, J=15.8, 6.6 Hz, =CHCH₂), 3.22 (2H, dd, J=6.6, 1.4 Hz, NCH₂CH=), 2.85-2.79 (2H, m, NCH₂CH₂Ph), 2.70-2.64 (2H, m, NCH₂CH₂Ph), 2.35 (3H, s, NCH₃). ¹³C NMR (101 MHz, CDCl₃) 140.5, 137.2, 132.6, 128.8, 128.6, 128.5, 127.5, 127.4, 126.4, 126.1, 60.3, 59.3, 42.3, 34.2.

***N*-Methyl-*N*-benzyl-allyl amine 8d**

The product was obtained as a colourless oil, yield: 81%, b.p.³: 80-82 °C/8 mmHg, ¹H NMR⁴ (270 MHz, CDCl₃) 7.16-7.32 (5H, m, ArH), 5.90 (1H, ddt, J=17.2, 10.5, 6.7 Hz, CH=CH₂), 5.18 (1H, ddt, J=17.2, 3.3, 1.5 Hz, CH=CHH(*cis*)), 5.12 (1H, ddt, J=10.5, 3.3, 1.2 Hz, CHCHH(*trans*)). 3.47 (2H, s, PhCH₂N), 3.05 (2H, ddd, J=6.7, 1.5, 1.2 Hz, NCH₂CH=), 2.17 (3H, s, NCH₃).

General Procedure for Preparation of Compounds 9a-c, 13 and 14

Et₂Zn (0.5mL, 0.5mmol, 1M solution in hexane) was diluted in Et₂O (2mL). To this solution was added CH₂I₂ (90μl 1mmol) at 0 °C. Twenty minutes after addition, the allylic amine derivative (0.455mmol in 0.5mL diethyl ether or DCM) was added. After 10 minutes, a white solid formed. The mixture was allowed to stir for 48 hours, then the solvent was

removed under reduced pressure and anhydrous THF (3mL) was added to re-dissolve the solid. The THF solution was cooled down to $-30\text{ }^{\circ}\text{C}$ before *n*-BuLi (1.1mL, 1.76mmol, 1.6M in hexane) was slowly added and the reactants were stirred for 1.5 hour at $-30\text{ }^{\circ}\text{C}$. After quenching the reaction with 10% EDTA (5mL) at room temperature, the product was extracted with petrol ($2 \times 30\text{mL}$). The organic solution was dried over Na_2SO_4 and concentrated to yield the pure product (for compounds **9a-c**) or the crude product (in the case of compound **13** and **14**; these compounds were purified by flash chromatography on silica gel).

***N*-But-3-enyl-methyl-phenethyl-amine 9a**

The product was obtained as colourless oil, yield: 70%, $\nu_{\text{max}}/\text{cm}^{-1}$ (neat) 3064, 3027, 2944, 2789, 1640, 1604, 1453. ^1H NMR (400 MHz, CDCl_3), 7.14-7.30 (5H, m, ArH), 5.81 (1H, ddt, $J=17.1, 10.2, 6.8\text{ Hz}$, $\text{CH}_2\text{CH=}$), 5.05 (1H, ddd, $J=17.1, 3.2, 1.5\text{ Hz}$), 4.99 (1H, ddd, $J=10.2, 3.2, 1.2\text{ Hz}$), 2.80-2.73 (2H, m, $\text{PhCH}_2\text{CH}_2\text{N}$), 2.65-2.57 (2H, m, $\text{PhCH}_2\text{CH}_2\text{N}$), 2.48 (2H, t, $J=7.6\text{ Hz}$, $=\text{CHCH}_2\text{CH}_2\text{N}$), 2.32 (3H, s, NCH_3), 2.29-2.19 (2H, m, $=\text{CHCH}_2\text{CH}_2\text{N}$). ^{13}C NMR (68 MHz, CDCl_3) 140.7, 136.9, 128.8, 128.4, 126.0, 115.6, 59.6, 57.1, 42.2, 34.0, 31.9. m/z (CI) 190 ($M+1$, 44), 148 (73), 98 (100). HRMS found: 190.1608, $\text{C}_{13}\text{H}_{20}\text{N}$ required 190.1596.

(2,2-Dimethyl-but-3-enyl)-methyl-phenethyl-amine 9b

The product was obtained as a colourless oil, yield: 50%, $\nu_{\text{max}}/\text{cm}^{-1}$ (neat) 3082, 3027, 2954, 2785, 1639, 1604, 1454. ^1H NMR (400 MHz, CDCl_3), 7.20 (5H, m, ArH), 5.85 (1H, dd, $J=17.6, 10.7\text{ Hz}$, CCH=), 4.93 (1H, dd, $J=17.6, 1.5\text{ Hz}$, $=\text{CHH}(cis)$), 4.91 (1H, dd, $J=10.7, 1.5\text{ Hz}$, $=\text{CHH}(trans)$), 2.78-2.70 (2H m, $\text{CH}_2\text{CH}_2\text{N}$), 2.67-2.61 (2H, m, $\text{CH}_2\text{CH}_2\text{N}$), 2.34 (3H, s, NCH_3), 2.27 (2H, s, NCH_2C), 1.05 (6H, s, $\text{C}(\text{CH}_3)_2$). ^{13}C NMR (101 MHz, CDCl_3)

148.0, 140.8, 128.9, 128.4, 125.9, 110.6, 69.4, 62.3, 45.2, 38.9, 34.3, 25.4. m/z (CI) 218 (M+1, 50), 216 (60), 148 (82), 84 (100). HRMS found: 218.1908, $C_{15}H_{24}N$ required 218.1909.

Methyl-phenethyl-(2-phenyl-but-3-enyl)-amine 9c

The product was obtained as colourless oil, yield: 76%, $\nu_{\max}/\text{cm}^{-1}$ (neat) 3062, 3026, 2943, 2788, 1636, 1602. ^1H NMR (400 MHz, CDCl_3) 7.12-7.34 (10H, m, ArH), 6.03 (1H, ddd, $J=17.6, 10.3, 7.8$ Hz, $=\text{CHCHPh}$), 5.08 (1H, dd, $J=10.3, 1.5$ Hz, $=\text{CHH}(\text{cis})$), 5.04 (1H, dd, $J=17.6, 1.5$ Hz, $=\text{CHH}(\text{trans})$), 3.53 (1H, dd, $J=15.2, 7.8$ Hz, NCHHCHPh), 2.80-2.56 (6H, m, $\text{CH}_2\text{CH}_2 + \text{NCHHCHPh}$), 2.32 (3H, s, NCH_3). ^{13}C NMR (101 MHz, CDCl_3) 143.1, 140.8, 140.6, 128.7, 128.4, 128.3, 127.8, 126.3, 125.9, 115.1, 62.8, 59.8, 48.2, 42.6, 33.4. m/z (CI) 266 (M+1, 8), 232 (40), 131 (56), 105 (100). HRMS found: 266.1906, $C_{19}H_{24}N$ required 266.1909.

(2R, 3R, 6S)-3,4-Dimethyl-2,6-diphenyl-3,4,5,6-tetrahydro-2H-[1,4]oxazocine 13 and (2R, 3R, 5R)-3,4-Dimethyl-2-phenyl-6-styryl-morpholine 14

(2R, 3R, 6S)-3,4-Dimethyl-2,6-diphenyl-3,4,5,6-tetrahydro-2H-[1,4] oxazocine compound **13** was purified by flash chromatography (10% ethyl acetate in petrol, $R_f=0.25$) and obtained as a white solid, yield: 72%, m.p.:101-102 °C, $[\alpha]_D^{20} -19$ ($c=0.39$, CHCl_3). $\nu_{\max}/\text{cm}^{-1}$ (CH_2Cl_2) 3032, 2960, 1647, 1600. ^1H NMR (400 MHz, CDCl_3) 7.20-7.41 (10H, m, ArH), 6.22 (1H, dd, $J=6.6, 1.7$ Hz, OCH=), 5.00-4.94 (2H, m, $\text{PhCHO} + \text{OCH=CH}$), 4.23 (1H, ddd, $J=11.7, 5.4, 4.4$ Hz, PhCHCH_2), 3.43 (1H, dq, $J=10.8, 6.4$ Hz, NCHCH_3), 3.15 (1H, dd, $J=13.2, 11.7$ Hz, NCHHCH), 3.03 (1H, dd, $J=13.2, 4.4$ Hz, NCHHCH), 2.61 (3H, s,

NCH₃), 0.92 (3H, d, J=6.4 Hz, NCHCH₃). ¹³C NMR (101 MHz, CDCl₃) 144.4, 143.8, 139.5, 128.7, 128.6, 128.3, 127.9, 127.7, 126.5, 87.0, 64.4, 61.9, 41.5, 38.6, 13.2. m/z (CI) 294 (M+1, 100), 187 (30), 130 (52). HRMS found: 294.1863, C₂₀H₂₄NO required 294.1858.

(2R, 3R, 5R)-3,4-Dimethyl-2-phenyl-6-styryl-morpholine compound **14** was purified by flash chromatography (2.5% methanol in DCM) and obtained as a pale yellow oil, yield: 7%. [α]_D²⁰ -15 (c=0.39, CHCl₃). $\epsilon_{\text{max}}/\text{cm}^{-1}$ (neat) 3061, 3028, 2789, 1450. ¹H NMR (400 MHz, CDCl₃) 7.19-7.41 (10H, m, ArH), 6.66 (1H, d, J=15.8 Hz, CH=CHPh), 6.21 (1H, dd, J=15.8, 6.1 Hz, CH=CHPh), 4.50-4.44 (1H, m, OCHCH₂), 4.23 (1H, d, J=9.3 Hz, PhCHO), 2.93 (1H, dd, J=11.2, 2.5 Hz, NCHHCH), 2.35 (1H, dd, J=11.2, 10.7 Hz, NCHHCH), 2.16 (1H, dq, J=9.3, 6.4 Hz, NCHCH₃), 0.87 (3H, d, J=6.4 Hz, NCHCH₃). ¹³C NMR (101 MHz, CDCl₃) 140.1, 136.9, 131.7, 128.5, 128.4, 128.3, 128.2, 127.7, 126.6, 85.0, 76.663.1, 61.3, 43.1, 15.0. m/z (CI) 294 (M+1, 73), 187 (47), 130 (100). HRMS found: 294.1860, C₂₀H₂₄NO required 294.1858.

3,4-Dimethyl-5-phenyl-2-styryl-oxazolidine **12**

(1R, 2R)-pseudo-Ephedrine (5mmol) and cinnamaldehyde (5mmol) were dissolved in DCM (20mL). To this solution was added Na₂SO₄ (5mmol). The mixture was stirred under N₂ overnight. After removing the desiccant, the solution was concentrated to obtain the crude product as a white solid which was further purified by recrystallization in hexane to yield the product, yield: 87%, m.p.: 74-5 °C, ¹H NMR⁵ (400 MHz, CDCl₃): 7.24-7.48 (10H, m, ArH), 6.75 (1H, d, J=16.1 Hz, PhCH=), 6.49 (1H, dd, J=16.1, 7.3 Hz, PhCH=CH), 4.65 (1H, d, J=8.8 Hz, PhCHO), 4.58 (1H, d, J=7.3 Hz, OCHN), 2.44 (1H, dq, J=8.8, 5.9 Hz, CH₃CH), 2.31 (3H, s, NCH₃), 1.19 (3H, d, J=5.9 Hz, CHCH₃).

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II X-ray data for compound 13

Crystallographic data are presented in Tables 1-5. A single crystal of compound **13** was coated in high-vacuum grease and mounted on a glass fibre. X-ray measurements were made using a Bruker SMART CCD area-detector diffractometer with Mo-K α radiation ($\lambda = 0.71073 \text{ \AA}$).¹ Intensities were integrated² from several series of exposures, each exposure covering 0.3° in θ , and the total data set being a sphere. Absorption corrections were applied, based on multiple and symmetry-equivalent measurements.³ The structure was solved by direct methods and refined by least squares on weighted F^2 values for all reflections (see Table 1).⁴

All non-hydrogen atoms were assigned anisotropic displacement parameters and refined without positional constraints. All other hydrogen atoms were constrained to ideal geometries and refined with isotropic displacement parameters equal to 1.5 times (methyl) or 1.2 times (all other hydrogens) the equivalent isotropic displacement parameter of their parent atom.

Refinement proceeded smoothly to give the residuals shown in Table 1. Complex neutral-atom scattering factors were used.⁵

References

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3. G. M. Sheldrick. *SADABS: A program for absorption correction with the Siemens SMART system*; University of Göttingen: Germany, 1996.
4. *SHELXTL program system version 5.1*; Bruker Analytical X-ray Instruments Inc., Madison, WI, 1998.
5. *International Tables for Crystallography*, Kluwer, Dordrecht, 1992, vol. C.

Table 1. Crystal data and structure refinement for compound **13**.

Identification code

compound **13**

Empirical formula	C ₂₀ H ₂₃ NO
Formula weight	293.39
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P2(1)2(1)2(1)
Unit cell dimensions	a = 6.3847(6) Å = 90° b = 11.9932(12) Å = 90° c = 21.566(2) Å = 90°
Volume	1651.4(3) Å ³
Z	4
Density (calculated)	1.180 Mg/m ³
Absorption coefficient	0.072 mm ⁻¹
F(000)	632
Crystal size	1.2 x 0.6 x 0.1 mm
range for data collection	1.89 to 27.53°
Index ranges	-8<=h<=8, -15<=k<=15, -28<=l<=28
Reflections collected	17315
Independent reflections	3799 [R _{int} = 0.0248]
Completeness to = 27.53°	99.7 %
Absorption correction	Multi-scan
Max. and min. transmission	0.991 and 0.778
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	3799 / 0 / 201
Goodness-of-fit on F ²	S = 1.010
R indices [for 3271 reflections with I>2 (I)]	R ₁ = 0.0332, wR ₂ = 0.0809
R indices (for all 3799 data)	R ₁ = 0.0402, wR ₂ = 0.0833
Weighting scheme	w ⁻¹ = ² (F _o ²) + (aP) ² , where P = [max(F _o ² , 0) + 2F _c ²]/3 a = 0.052
Largest diff. peak and hole	0.130 and -0.173 eÅ ⁻³

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for compound **13**. U(eq) is defined as one third of the trace of the

orthogonalized U_{ij} tensor.				
	x	y	z	U(eq)
C(1)	8969(2)	3985(1)	3021(1)	32(1)
C(2)	6749(2)	4459(1)	3106(1)	31(1)
C(3)	4675(2)	4697(1)	2127(1)	33(1)
C(4)	5707(2)	3957(1)	1618(1)	32(1)
C(5)	6289(2)	2829(1)	1875(1)	33(1)
C(6)	7872(2)	2657(1)	2259(1)	34(1)
C(7)	6653(2)	5090(1)	3726(1)	40(1)
C(8)	7498(2)	5995(1)	2369(1)	42(1)
C(9)	9655(2)	3189(1)	3526(1)	34(1)
C(10)	8402(2)	2310(1)	3728(1)	40(1)
C(11)	9116(3)	1573(1)	4177(1)	52(1)
C(12)	11066(3)	1718(2)	4435(1)	61(1)
C(13)	12311(3)	2588(2)	4247(1)	61(1)
C(14)	11618(2)	3318(1)	3794(1)	46(1)
C(15)	4229(2)	3851(1)	1067(1)	33(1)
C(16)	4806(2)	4202(1)	479(1)	40(1)
C(17)	3428(3)	4101(1)	-17(1)	50(1)
C(18)	1460(3)	3647(1)	67(1)	46(1)
C(19)	867(2)	3301(1)	652(1)	42(1)
C(20)	2229(2)	3405(1)	1147(1)	37(1)
N(1)	6007(2)	5178(1)	2604(1)	32(1)
O(1)	9247(1)	3501(1)	2410(1)	35(1)

Table 3. Bond lengths [$\text{\AA}$] and angles [$^\circ$] for compound **13**.

C(1)-O(1)	1.4514(14)
C(1)-C(9)	1.5133(18)
C(1)-C(2)	1.5383(18)
C(1)-H(1)	1.0000
C(2)-N(1)	1.4639(15)
C(2)-C(7)	1.5360(16)
C(2)-H(2)	1.0000
C(3)-N(1)	1.4539(15)
C(3)-C(4)	1.5574(16)
C(3)-H(3A)	0.9900
C(3)-H(3B)	0.9900
C(4)-C(5)	1.5087(17)
C(4)-C(15)	1.5229(17)
C(4)-H(4)	1.0000
C(5)-C(6)	1.3215(18)
C(5)-H(5)	0.9500
C(6)-O(1)	1.3793(15)
C(6)-H(6)	0.9500
C(7)-H(7A)	0.9800
C(7)-H(7B)	0.9800
C(7)-H(7C)	0.9800
C(8)-N(1)	1.4564(16)
C(8)-H(8A)	0.9800
C(8)-H(8B)	0.9800
C(8)-H(8C)	0.9800
C(9)-C(14)	1.3878(19)
C(9)-C(10)	1.3930(19)
C(10)-C(11)	1.3881(19)
C(10)-H(10)	0.9500
C(11)-C(12)	1.375(2)
C(11)-H(11)	0.9500
C(12)-C(13)	1.373(3)
C(12)-H(12)	0.9500
C(13)-C(14)	1.385(2)
C(13)-H(13)	0.9500
C(14)-H(14)	0.9500
C(15)-C(16)	1.3858(17)
C(15)-C(20)	1.3952(19)

C(16)-C(17)	1.391(2)
C(16)-H(16)	0.9500
C(17)-C(18)	1.381(2)
C(17)-H(17)	0.9500
C(18)-C(19)	1.382(2)
C(18)-H(18)	0.9500
C(19)-C(20)	1.3825(18)
C(19)-H(19)	0.9500
C(20)-H(20)	0.9500
O(1)-C(1)-C(9)	111.46(10)
O(1)-C(1)-C(2)	111.63(9)
C(9)-C(1)-C(2)	114.45(10)
O(1)-C(1)-H(1)	106.2
C(9)-C(1)-H(1)	106.2
C(2)-C(1)-H(1)	106.2
N(1)-C(2)-C(7)	109.89(10)
N(1)-C(2)-C(1)	115.30(9)
C(7)-C(2)-C(1)	108.78(10)
N(1)-C(2)-H(2)	107.5
C(7)-C(2)-H(2)	107.5
C(1)-C(2)-H(2)	107.5
N(1)-C(3)-C(4)	118.48(10)
N(1)-C(3)-H(3A)	107.7
C(4)-C(3)-H(3A)	107.7
N(1)-C(3)-H(3B)	107.7
C(4)-C(3)-H(3B)	107.7
H(3A)-C(3)-H(3B)	107.1
C(5)-C(4)-C(15)	111.36(10)
C(5)-C(4)-C(3)	110.87(9)
C(15)-C(4)-C(3)	109.61(10)
C(5)-C(4)-H(4)	108.3
C(15)-C(4)-H(4)	108.3
C(3)-C(4)-H(4)	108.3
C(6)-C(5)-C(4)	123.96(12)
C(6)-C(5)-H(5)	118.0
C(4)-C(5)-H(5)	118.0
C(5)-C(6)-O(1)	121.32(11)

C(5)-C(6)-H(6)	119.3
O(1)-C(6)-H(6)	119.3
C(2)-C(7)-H(7A)	109.5
C(2)-C(7)-H(7B)	109.5
H(7A)-C(7)-H(7B)	109.5
C(2)-C(7)-H(7C)	109.5
H(7A)-C(7)-H(7C)	109.5
H(7B)-C(7)-H(7C)	109.5
N(1)-C(8)-H(8A)	109.5
N(1)-C(8)-H(8B)	109.5
H(8A)-C(8)-H(8B)	109.5
N(1)-C(8)-H(8C)	109.5
H(8A)-C(8)-H(8C)	109.5
H(8B)-C(8)-H(8C)	109.5
C(14)-C(9)-C(10)	118.24(13)
C(14)-C(9)-C(1)	119.36(12)
C(10)-C(9)-C(1)	122.38(11)
C(11)-C(10)-C(9)	120.72(14)
C(11)-C(10)-H(10)	119.6
C(9)-C(10)-H(10)	119.6
C(12)-C(11)-C(10)	119.94(16)
C(12)-C(11)-H(11)	120.0
C(10)-C(11)-H(11)	120.0
C(13)-C(12)-C(11)	120.09(15)
C(13)-C(12)-H(12)	120.0
C(11)-C(12)-H(12)	120.0
C(12)-C(13)-C(14)	120.22(15)
C(12)-C(13)-H(13)	119.9
C(14)-C(13)-H(13)	119.9
C(13)-C(14)-C(9)	120.78(15)
C(13)-C(14)-H(14)	119.6
C(9)-C(14)-H(14)	119.6
C(16)-C(15)-C(20)	118.24(12)
C(16)-C(15)-C(4)	121.58(12)
C(20)-C(15)-C(4)	120.18(10)
C(15)-C(16)-C(17)	120.61(14)
C(15)-C(16)-H(16)	119.7
C(17)-C(16)-H(16)	119.7

C(18)-C(17)-C(16)	120.58(12)
C(18)-C(17)-H(17)	119.7
C(16)-C(17)-H(17)	119.7
C(17)-C(18)-C(19)	119.21(13)
C(17)-C(18)-H(18)	120.4
C(19)-C(18)-H(18)	120.4
C(18)-C(19)-C(20)	120.36(14)
C(18)-C(19)-H(19)	119.8
C(20)-C(19)-H(19)	119.8
C(19)-C(20)-C(15)	120.99(12)
C(19)-C(20)-H(20)	119.5
C(15)-C(20)-H(20)	119.5
C(3)-N(1)-C(8)	113.81(10)
C(3)-N(1)-C(2)	118.59(10)
C(8)-N(1)-C(2)	116.25(10)
C(6)-O(1)-C(1)	115.55(9)

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound **13**.

The anisotropic displacement factor exponent takes the form:

$$-2 \text{ }^2 [h^2 \text{ a}^*{}^2 U_{11} + \dots + 2 h k \text{ a}^* \text{ b}^* U_{12}]$$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	29(1)	34(1)	33(1)	-3(1)	3(1)	-7(1)
C(2)	33(1)	29(1)	30(1)	-1(1)	5(1)	-5(1)
C(3)	36(1)	32(1)	32(1)	-1(1)	1(1)	2(1)
C(4)	34(1)	32(1)	30(1)	-1(1)	3(1)	-2(1)
C(5)	38(1)	31(1)	31(1)	-4(1)	3(1)	1(1)
C(6)	36(1)	32(1)	33(1)	-2(1)	5(1)	3(1)
C(7)	49(1)	38(1)	33(1)	-4(1)	3(1)	-3(1)
C(8)	55(1)	32(1)	39(1)	3(1)	5(1)	-9(1)
C(9)	32(1)	40(1)	31(1)	-8(1)	0(1)	1(1)
C(10)	45(1)	38(1)	35(1)	0(1)	-4(1)	0(1)
C(11)	73(1)	45(1)	38(1)	2(1)	2(1)	11(1)
C(12)	75(1)	72(1)	34(1)	-2(1)	-5(1)	35(1)
C(13)	46(1)	97(1)	39(1)	-16(1)	-13(1)	25(1)
C(14)	34(1)	66(1)	38(1)	-14(1)	0(1)	3(1)
C(15)	40(1)	28(1)	30(1)	-2(1)	1(1)	5(1)
C(16)	48(1)	39(1)	34(1)	2(1)	6(1)	2(1)
C(17)	74(1)	48(1)	27(1)	4(1)	3(1)	6(1)
C(18)	61(1)	44(1)	35(1)	-4(1)	-12(1)	10(1)
C(19)	43(1)	41(1)	43(1)	-4(1)	-6(1)	3(1)
C(20)	43(1)	36(1)	31(1)	0(1)	1(1)	0(1)
N(1)	38(1)	28(1)	30(1)	-1(1)	2(1)	-4(1)
O(1)	30(1)	44(1)	31(1)	-3(1)	6(1)	-2(1)

Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^{-3}$) for compound **13**.

	x	y	z	U(eq)
H(1)	9944	4637	3043	38
H(2)	5764	3812	3136	37
H(3A)	3942	5317	1916	40
H(3B)	3592	4243	2337	40
H(4)	7016	4335	1475	38
H(5)	5468	2205	1755	40
H(6)	8067	1937	2433	41
H(7A)	5231	5374	3791	59
H(7B)	7642	5714	3716	59
H(7C)	7026	4583	4064	59
H(8A)	6763	6538	2108	63
H(8B)	8573	5615	2123	63
H(8C)	8161	6381	2718	63
H(10)	7045	2213	3556	47
H(11)	8258	969	4306	62
H(12)	11553	1215	4743	73
H(13)	13654	2689	4429	73
H(14)	12495	3914	3664	55
H(16)	6156	4515	415	48
H(17)	3844	4346	-418	59
H(18)	526	3575	-273	56
H(19)	-485	2989	715	51
H(20)	1797	3169	1548	44