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;Department of Chemistry and Biochemistry
  Duquesne University
  Pittsburgh, PA 15282 USA
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;The crystal structure of (+-)-(1R)-3-cyano-
1-methyl-2-phenylpropyl)4-nitrobenzoate (8a
4-nitrobenzoate ester)is reported as supporting
information in the article entitled, Hydroxy
Alkenenitriles - Diastereoselective Conjugate
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for publication. The asymmetric unit consists
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;Fraser F. Fleming
;
;Department of Chemistry and Biochemistry
  Duquesne University
  Pittsburgh, PA 15282 USA
;
;Qunzhao Wang
;
;Department of Chemistry and Biochemistry
  Duquesne University
  Pittsburgh, PA 15282 USA
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;Omar W.. Steward
;
;Department of Chemistry and Biochemistry
  Duquesne University

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Pittsburgh, PA 15282 USA

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Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations

;

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;Chelation-controlled conjugation addition of Grignard reagents to cyclic and acyclic β -hydroxyalkenenitriles stereoselectively generates β -substituted hydroxynitriles. *t*-BuMgCl-initiated deprotonation and chloride-alkyl exchange from a second Grignard reagent, generates an alkylmagnesium alkoxide that triggers conjugate and generates a metallated nitrile. Sequential conjugate addition-alkylation with alkyl halide and carbonyl electrophiles efficiently installs two new bonds and up to three stereocenters in a single synthetic operation.

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ENTER EXPERIMENTAL SECTION

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;The crystal structure of (+-)-(1*R*)-3-cyano-1-methyl-2-phenylpropyl)4-nitrobenzoate (9a 4-nitrobenzoate ester) is reported as supporting information in the article entitled, Hydroxy Alkenenitriles - Diastereoselective Conjugate Addition-Alkylations, which has been submitted to the Journal of Organic Chemistry for publication. The asymmetric unit consists of a single molecule.

;

_publ_section_references

;

ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan. Single Crystal Structure Analysis Software. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968). Acta Cryst. A24, 351-359.

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;The compound (+-)-(1R)-3-cyano-1-methyl-
2-phenylpropyl)4-nitrobenzoate
(8a 4-nitrobenzoate ester) was
synthesized as described in the paper.
The compound (8a 4-nitrobenzoate ester)
was crystallized from 1:2
EtOAc/hexanes, m.p. 377-380 K

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\y scans (North,Phillips & Matthews, 1968)

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(up to 4 scans to achieve I/s(I) > 3).
Stationary background counts were recorded at each end of the
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 Pittsburgh, PA 15282 USA

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 4-nitrobenzoate (12a di-4-nitrobenzoate ester) is reported
 as supporting information in the article entitled,
 Hydroxy Alkenenitriles - Diastereoselective Conjugate
 Addition-Alkylations, which has been submitted to the

Journal of Organic Chemistry for publication.
The asymmetric unit consists of a single molecule.

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;
;Department of Chemistry and Biochemistry
Duquesne University
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;
;Department of Chemistry and Biochemistry
Duquesne University
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Hydroxy Alkenenitriles - Diastereoselective Conjugate
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reagents to cyclic and acyclic γ -hydroxyalkenenitriles
stereoselectively generates β -substituted hydroxynitriles.
t-BuMgCl-initiated deprotonation and chloride-alkyl
exchange from a second Grignard reagent, generates an
alkylmagnesium alkoxide that triggers conjugate and
generates a metallated nitrile. Sequential conjugate
addition-alkylation with alkyl halide and carbonyl
electrophiles efficiently installs two new bonds and
up to three stereocenters in a single synthetic operation.
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as supporting information in the article entitled,
Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations, which has been submitted to the
Journal of Organic Chemistry for publication.
The asymmetric unit consists of a single molecule.
;

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  ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968).
Acta Cryst. A24, 351-359.
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 1-methyl-4-(4-nitrophenylcarbonyloxy)-
 2,4-diphenylbutyl 4-nitrobenzoate
 (12a di-4-nitrobenzoate ester) was
 synthesized as described in the paper.
 The compound (12a di-4-nitrobenzoate ester)
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\y scans (North,Phillips & Matthews, 1968)
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O(1)	0.049(3)	0.059(3)	0.086(3)	0.002(2)	-0.016(3)	0.007(2)
O(2)	0.124(5)	0.059(3)	0.160(6)	-0.001(3)	-0.057(5)	-0.009(4)
O(3)	0.073(6)	0.29(1)	0.198(9)	-0.019(7)	-0.036(6)	0.12(1)
O(4)	0.128(6)	0.31(1)	0.132(6)	0.127(8)	0.001(5)	0.076(7)
O(5)	0.044(2)	0.058(2)	0.070(3)	0.006(2)	-0.005(2)	-0.010(3)
O(6)	0.092(4)	0.070(3)	0.074(3)	0.016(3)	-0.028(3)	-0.012(3)
O(7)	0.137(5)	0.113(5)	0.126(6)	0.047(4)	-0.047(5)	0.004(5)
O(8)	0.224(9)	0.143(6)	0.124(6)	0.124(6)	-0.003(6)	-0.002(5)
N(1)	0.047(4)	0.083(4)	0.094(5)	0.009(3)	0.004(3)	0.000(4)
N(2)	0.065(7)	0.27(2)	0.095(7)	0.032(8)	0.011(5)	0.097(9)
N(3)	0.119(6)	0.071(6)	0.097(6)	0.036(5)	0.009(5)	0.011(5)
C(1)	0.047(4)	0.070(5)	0.054(4)	-0.002(3)	-0.013(3)	-0.007(3)
C(2)	0.039(4)	0.046(4)	0.059(4)	-0.001(3)	-0.010(3)	-0.002(3)
C(3)	0.029(3)	0.051(4)	0.062(4)	0.006(3)	-0.004(3)	-0.001(3)
C(4)	0.034(3)	0.060(4)	0.082(5)	-0.004(3)	-0.007(3)	-0.001(4)
C(5)	0.054(4)	0.072(5)	0.102(6)	-0.003(4)	0.017(4)	0.008(5)
C(6)	0.045(4)	0.059(4)	0.060(4)	-0.010(4)	0.005(4)	-0.008(4)
C(7)	0.064(4)	0.060(4)	0.054(4)	-0.002(4)	-0.008(4)	-0.001(3)
C(8)	0.066(5)	0.126(7)	0.073(6)	-0.015(5)	-0.004(5)	-0.022(5)
C(9)	0.114(8)	0.17(1)	0.058(5)	-0.010(7)	0.000(6)	-0.026(6)
C(10)	0.098(7)	0.147(9)	0.076(7)	-0.003(7)	0.021(6)	0.005(6)
C(11)	0.066(5)	0.108(7)	0.078(6)	-0.019(5)	0.005(5)	0.019(5)
C(12)	0.058(4)	0.076(5)	0.060(4)	-0.008(4)	-0.005(4)	0.000(4)
C(13)	0.042(3)	0.056(4)	0.060(4)	-0.003(3)	0.007(4)	-0.009(3)
C(14)	0.044(4)	0.070(4)	0.074(5)	0.002(4)	-0.003(4)	-0.014(4)
C(15)	0.057(4)	0.116(7)	0.071(5)	-0.004(5)	0.002(4)	-0.027(5)
C(16)	0.108(7)	0.097(7)	0.076(6)	-0.030(6)	0.014(6)	-0.043(5)
C(17)	0.125(7)	0.071(5)	0.079(6)	0.002(5)	0.007(6)	-0.024(5)
C(18)	0.087(6)	0.066(5)	0.060(5)	0.005(4)	0.007(4)	-0.007(4)
C(19)	0.074(5)	0.061(5)	0.077(5)	0.010(4)	-0.002(4)	0.009(4)
C(20)	0.062(4)	0.080(6)	0.066(5)	0.025(5)	-0.004(4)	0.017(4)
C(21)	0.083(6)	0.082(6)	0.088(6)	0.020(5)	0.004(5)	0.018(5)

C(22)	0.083(7)	0.129(9)	0.082(6)	0.046(6)	0.021(5)	0.047(6)
C(23)	0.049(5)	0.159(9)	0.075(6)	0.031(6)	0.010(4)	0.067(7)
C(24)	0.059(5)	0.119(7)	0.088(6)	0.000(5)	-0.008(5)	0.036(5)
C(25)	0.064(5)	0.091(6)	0.087(6)	0.005(5)	-0.003(5)	0.026(5)
C(26)	0.062(5)	0.042(4)	0.065(5)	-0.003(3)	-0.002(4)	-0.002(4)
C(27)	0.063(4)	0.037(3)	0.050(4)	0.000(3)	-0.004(4)	-0.007(3)
C(28)	0.092(5)	0.067(5)	0.077(5)	0.032(4)	-0.020(4)	-0.020(4)
C(29)	0.108(6)	0.091(6)	0.066(5)	0.017(5)	-0.024(5)	-0.010(5)
C(30)	0.072(5)	0.051(4)	0.064(5)	0.015(4)	0.004(4)	0.011(4)
C(31)	0.124(6)	0.056(4)	0.067(5)	0.042(5)	0.008(5)	0.007(4)
C(32)	0.112(6)	0.070(5)	0.054(4)	0.008(5)	-0.013(4)	-0.008(4)

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_refine_ls_extinction_method	none
_refine_ls_extinction_coef	?
_refine_ls_abs_structure_details	?
_refine_ls_abs_structure_Flack	?
_refine_ls_number_reflns	1257
_refine_ls_number_parameters	388
_refine_ls_number_restraints	0
_refine_ls_number_constraints	0
_refine_ls_R_factor_all	?
_refine_ls_R_factor_gt	0.0342
_refine_ls_wR_factor_all	?
_refine_ls_wR_factor_ref	0.0403
_refine_ls_goodness_of_fit_all	?
_refine_ls_goodness_of_fit_ref	1.305
_refine_ls_shift/su_max	0.0018
_refine_ls_shift/su_mean	0.0000
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loop_

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_geom_bond_distance

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_geom_bond_site_symmetry_2

_geom_bond_publ_flag

O(1) C(1) 1.440(7) . . yes
O(1) C(19) 1.351(8) . . yes
O(2) C(19) 1.179(8) . . yes
O(3) N(2) 1.21(2) . . yes
O(4) N(2) 1.25(1) . . yes
O(5) C(4) 1.460(7) . . yes
O(5) C(26) 1.332(7) . . yes
O(6) C(26) 1.202(7) . . yes
O(7) N(3) 1.189(8) . . yes
O(8) N(3) 1.199(9) . . yes
N(1) C(6) 1.155(7) . . yes
N(2) C(23) 1.51(1) . . yes
N(3) C(30) 1.479(9) . . yes
C(1) C(2) 1.548(8) . . yes
C(1) C(7) 1.503(9) . . yes
C(1) H(1) 0.96 . . no
C(2) C(3) 1.548(8) . . yes
C(2) C(6) 1.452(9) . . yes
C(2) H(2) 0.95 . . no
C(3) C(4) 1.539(8) . . yes
C(3) C(13) 1.504(8) . . yes
C(3) H(3) 0.95 . . no
C(4) C(5) 1.513(9) . . yes
C(4) H(4) 0.95 . . no
C(5) H(5) 0.96 . . no
C(5) H(6) 0.96 . . no
C(5) H(7) 0.95 . . no
C(7) C(8) 1.372(9) . . yes
C(7) C(12) 1.418(9) . . yes
C(8) C(9) 1.37(1) . . yes
C(8) H(8) 0.95 . . no
C(9) C(10) 1.35(1) . . yes
C(9) H(9) 0.97 . . no
C(10) C(11) 1.38(1) . . yes
C(10) H(10) 0.95 . . no
C(11) C(12) 1.37(1) . . yes
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C(12) H(12) 0.95 . . no
C(13) C(14) 1.372(8) . . yes
C(13) C(18) 1.398(9) . . yes
C(14) C(15) 1.391(9) . . yes
C(14) H(13) 0.96 . . no
C(15) C(16) 1.38(1) . . yes
C(15) H(14) 0.97 . . no
C(16) C(17) 1.35(1) . . yes
C(16) H(15) 0.95 . . no
C(17) C(18) 1.38(1) . . yes
C(17) H(16) 0.96 . . no
C(18) H(17) 0.95 . . no
C(19) C(20) 1.50(1) . . yes
C(20) C(21) 1.395(9) . . yes

C(20) C(25) 1.40(1) . . yes
 C(21) C(22) 1.40(1) . . yes
 C(21) H(18) 0.95 . . no
 C(22) C(23) 1.37(1) . . yes
 C(22) H(19) 0.96 . . no
 C(23) C(24) 1.35(1) . . yes
 C(24) C(25) 1.38(1) . . yes
 C(24) H(20) 0.96 . . no
 C(25) H(21) 0.96 . . no
 C(26) C(27) 1.475(9) . . yes
 C(27) C(28) 1.355(9) . . yes
 C(27) C(32) 1.379(9) . . yes
 C(28) C(29) 1.391(9) . . yes
 C(28) H(22) 0.95 . . no
 C(29) C(30) 1.36(1) . . yes
 C(29) H(23) 0.96 . . no
 C(30) C(31) 1.340(9) . . yes
 C(31) C(32) 1.373(9) . . yes
 C(31) H(24) 0.95 . . no
 C(32) H(25) 0.94 . . no
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 C(1) O(1) C(19) 117.6(5) . . . yes
 C(4) O(5) C(26) 118.6(5) . . . yes
 O(3) N(2) O(4) 128(1) . . . yes
 O(3) N(2) C(23) 118(1) . . . yes
 O(4) N(2) C(23) 114(1) . . . yes
 O(7) N(3) O(8) 124.8(8) . . . yes
 O(7) N(3) C(30) 118.8(8) . . . yes
 O(8) N(3) C(30) 116.2(8) . . . yes
 O(1) C(1) C(2) 102.9(4) . . . yes
 O(1) C(1) C(7) 110.5(5) . . . yes
 O(1) C(1) H(1) 110.0 . . . no
 C(2) C(1) C(7) 114.0(5) . . . yes
 C(2) C(1) H(1) 108.8 . . . no
 C(7) C(1) H(1) 110.3 . . . no
 C(1) C(2) C(3) 111.8(5) . . . yes
 C(1) C(2) C(6) 110.1(5) . . . yes
 C(1) C(2) H(2) 107.7 . . . no
 C(3) C(2) C(6) 112.1(5) . . . yes
 C(3) C(2) H(2) 107.0 . . . no
 C(6) C(2) H(2) 108.0 . . . no
 C(2) C(3) C(4) 112.4(5) . . . yes

C(2) C(3) C(13) 113.9(5) . . . yes
C(2) C(3) H(3) 105.0 . . . no
C(4) C(3) C(13) 116.2(5) . . . yes
C(4) C(3) H(3) 104.3 . . . no
C(13) C(3) H(3) 103.5 . . . no
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O(5) C(4) C(5) 106.4(5) . . . yes
O(5) C(4) H(4) 109.6 . . . no
C(3) C(4) C(5) 112.9(5) . . . yes
C(3) C(4) H(4) 109.0 . . . no
C(5) C(4) H(4) 107.8 . . . no
C(4) C(5) H(5) 110.6 . . . no
C(4) C(5) H(6) 110.2 . . . no
C(4) C(5) H(7) 110.8 . . . no
H(5) C(5) H(6) 107.9 . . . no
H(5) C(5) H(7) 108.7 . . . no
H(6) C(5) H(7) 108.5 . . . no
N(1) C(6) C(2) 177.1(7) . . . yes
C(1) C(7) C(8) 122.4(6) . . . yes
C(1) C(7) C(12) 119.6(6) . . . yes
C(8) C(7) C(12) 118.0(6) . . . yes
C(7) C(8) C(9) 120.3(7) . . . yes
C(7) C(8) H(8) 119.5 . . . no
C(9) C(8) H(8) 120.2 . . . no
C(8) C(9) C(10) 121.7(8) . . . yes
C(8) C(9) H(9) 119.2 . . . no
C(10) C(9) H(9) 119.1 . . . no
C(9) C(10) C(11) 119.7(8) . . . yes
C(9) C(10) H(10) 120.4 . . . no
C(11) C(10) H(10) 119.9 . . . no
C(10) C(11) C(12) 119.7(7) . . . yes
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C(12) C(11) H(11) 119.6 . . . no
C(7) C(12) C(11) 120.5(6) . . . yes
C(7) C(12) H(12) 119.3 . . . no
C(11) C(12) H(12) 120.2 . . . no
C(3) C(13) C(14) 122.8(5) . . . yes
C(3) C(13) C(18) 119.6(6) . . . yes
C(14) C(13) C(18) 117.6(6) . . . yes
C(13) C(14) C(15) 120.8(6) . . . yes
C(13) C(14) H(13) 119.8 . . . no
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C(14) C(15) C(16) 120.1(8) . . . yes
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C(15) C(16) C(17) 119.8(7) . . . yes
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C(17) C(16) H(15) 119.4 . . . no
C(16) C(17) C(18) 120.3(7) . . . yes
C(16) C(17) H(16) 120.7 . . . no
C(18) C(17) H(16) 119.0 . . . no

C(13) C(18) C(17) 121.2(7) . . . yes
 C(13) C(18) H(17) 118.7 . . . no
 C(17) C(18) H(17) 120.1 . . . no
 O(1) C(19) O(2) 123.7(7) . . . yes
 O(1) C(19) C(20) 109.6(7) . . . yes
 O(2) C(19) C(20) 126.8(7) . . . yes
 C(19) C(20) C(21) 117.2(7) . . . yes
 C(19) C(20) C(25) 122.3(7) . . . yes
 C(21) C(20) C(25) 120.5(7) . . . yes
 C(20) C(21) C(22) 119.5(8) . . . yes
 C(20) C(21) H(18) 118.6 . . . no
 C(22) C(21) H(18) 121.9 . . . no
 C(21) C(22) C(23) 117.6(8) . . . yes
 C(21) C(22) H(19) 117.3 . . . no
 C(23) C(22) H(19) 125.1 . . . no
 N(2) C(23) C(22) 119(1) . . . yes
 N(2) C(23) C(24) 116(1) . . . yes
 C(22) C(23) C(24) 124.4(8) . . . yes
 C(23) C(24) C(25) 118.9(8) . . . yes
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 C(25) C(24) H(20) 120.8 . . . no
 C(20) C(25) C(24) 119.2(7) . . . yes
 C(20) C(25) H(21) 119.6 . . . no
 C(24) C(25) H(21) 121.2 . . . no
 O(5) C(26) O(6) 123.7(6) . . . yes
 O(5) C(26) C(27) 112.0(6) . . . yes
 O(6) C(26) C(27) 124.2(6) . . . yes
 C(26) C(27) C(28) 122.5(6) . . . yes
 C(26) C(27) C(32) 118.5(6) . . . yes
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 C(29) C(30) C(31) 122.1(7) . . . yes
 C(30) C(31) C(32) 118.8(7) . . . yes
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loop_

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_geom_contact_atom_site_label_2

_geom_contact_distance

_geom_contact_site_symmetry_1


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_geom_contact_site_symmetry_2
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O(2)   O(4)    3.30(1)   . 4_455 no
O(3)   C(10)   3.29(1)   . 1_655 no
O(3)   C(11)   3.36(1)   . 1_655 no
O(3)   O(7)    3.51(1)   . 3_545 no
O(3)   C(28)   3.54(1)   . 2_665 no
O(4)   O(7)    3.17(1)   . 3_545 no
O(4)   C(17)   3.27(1)   . 2_655 no
O(4)   C(16)   3.35(1)   . 2_655 no
O(4)   C(21)   3.54(1)   . 4 no
O(5)   O(8)    3.207(8)  . 4_575 no
O(6)   O(8)    3.14(1)   . 4_575 no
O(6)   C(11)   3.430(9)  . 4_565 no
O(7)   N(2)    3.06(1)   . 3_564 no
O(7)   C(23)   3.21(1)   . 3_564 no
O(7)   C(22)   3.37(1)   . 3_564 no
O(8)   C(26)   3.04(1)   . 4_475 no
O(8)   C(4)    3.34(1)   . 4_475 no
O(8)   C(5)    3.36(1)   . 4_475 no
N(1)   C(4)    3.328(8)  . 4_465 no
N(1)   C(5)    3.38(1)   . 4_465 no
N(1)   C(25)   3.52(1)   . 4_465 no
N(1)   C(3)    3.577(8)  . 4_465 no
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O(2) C(19) C(20) C(21) . . . . -5(1) no
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O(4) N(2) C(23) C(24) . . . . -173.6(9) no
O(5) C(4) C(3) C(2) . . . . -57.7(6) no

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 O(6) C(26) O(5) C(4) -9.6(9) no
 O(6) C(26) C(27) C(28) -179.8(7) no
 O(6) C(26) C(27) C(32) 1(1) no
 O(7) N(3) C(30) C(29) 2(1) no
 O(7) N(3) C(30) C(31) 178.5(8) no
 O(8) N(3) C(30) C(29) -172.0(9) no
 O(8) N(3) C(30) C(31) 4(1) no
 N(1) C(6) C(2) C(1) 87.6(1) no
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;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

_publ_contact_letter

;The crystal structure of (+-)-(2R,4R)-4-hydroxy-3-phenyl-

2-benzylpentanenitrile (12b) is reported as supporting

information in the article entitled, Hydroxy

Alkenenitriles - Diastereoselective Conjugate

Addition-Alkylations, which has been submitted

to the Journal of Organic Chemistry for publication.

The asymmetric unit consists of a single molecule.

An intermolecular hydrogen bond is observed between

the hydroxyl and nitrile groups: O(1)-N(2) 2.944(3) Å.

;

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_publ_contact_author_phone '(412) 396 6338 '

_publ_contact_author_fax '(412) 396 5683 '

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_publ_author_name

_publ_author_address

;Fraser F. Fleming

;

;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

;Qunzhao Wang

;

;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

;Omar W.. Steward

;

;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

_publ_section_title

;

Hydroxy Alkenenitriles - Diastereoselective Conjugate

Addition-Alkylations

;

_publ_section_title_footnote 'ENTER ANY FOOTNOTES TO TITLE'

_publ_section_abstract

;Chelation-controlled conjugation addition of Grignard reagents to cyclic and acyclic γ -hydroxyalkenenitriles stereoselectively generates β -substituted hydroxynitriles. t-BuMgCl-initiated deprotonation and chloride-alkyl exchange from a second Grignard reagent, generates an alkylmagnesium alkoxide that triggers conjugate and generates a metallated nitrile. Sequential conjugate addition-alkylation with alkyl halide and carbonyl electrophiles efficiently installs two new bonds and up to three stereocenters in a single synthetic operation.

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ENTER EXPERIMENTAL SECTION

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; The crystal structure of (+)-(2R,4R)-4-hydroxy-3-phenyl-2-benzylpentanenitrile (12b) is reported as supporting information in the article entitled, Hydroxy Alkenenitriles - Diastereoselective Conjugate Addition-Alkylations, which has been submitted to the Journal of the American Society for publication. The asymmetric unit consists of a single molecule. An intermolecular hydrogen bond is observed between the hydroxyl and nitrile groups: O(1)-N(2) 2.944(3) Å.

;

_publ_section_references

;

ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan. Single Crystal Structure Analysis Software. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968). Acta Cryst. A24, 351-359.

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ENTER FIGURE CAPTIONS

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The compound,(+)-(2R,4R)-4-hydroxy-3-phenyl-2-
-benzylpentanenitrile (12b) was synthesized
as described in the paper. The compound 12b
was crystallized from 1:2 EtOAc/hexanes,
m.p. 380-385 K.
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\y scans (North,Phillips & Matthews, 1968)

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Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

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C(12) 0.6611(3) 0.1323(2) 1.1550(4) 0.0768(9) 1.000 . Uani d ?
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C(2)	0.073(2)	0.045(1)	0.064(1)	-0.004(1)	-0.006(1)	-0.003(1)
C(3)	0.060(1)	0.053(1)	0.050(1)	-0.002(1)	0.003(1)	-0.007(1)
C(4)	0.057(1)	0.048(1)	0.053(1)	-0.005(1)	0.006(1)	0.0027(9)
C(5)	0.062(1)	0.064(1)	0.073(2)	-0.004(1)	0.011(1)	0.016(1)
C(6)	0.057(1)	0.058(1)	0.072(2)	-0.003(1)	0.003(1)	0.013(1)
C(7)	0.059(1)	0.051(1)	0.067(1)	-0.014(1)	0.006(1)	0.016(1)
C(8)	0.078(2)	0.055(1)	0.081(2)	-0.006(1)	-0.007(1)	0.006(1)
C(9)	0.096(2)	0.054(1)	0.102(2)	-0.002(1)	0.011(2)	0.002(1)
C(10)	0.074(2)	0.066(2)	0.117(3)	0.002(1)	0.004(2)	0.023(2)
C(11)	0.074(2)	0.092(2)	0.100(2)	-0.008(2)	-0.017(2)	0.021(2)
C(12)	0.081(2)	0.076(2)	0.073(2)	-0.009(1)	-0.001(1)	0.006(1)
C(13)	0.055(1)	0.051(1)	0.052(1)	0.000(1)	0.002(1)	0.000(1)
C(14)	0.065(1)	0.064(1)	0.060(1)	-0.009(1)	0.007(1)	-0.007(1)
C(15)	0.077(2)	0.069(2)	0.076(2)	-0.015(1)	-0.003(1)	-0.008(1)
C(16)	0.060(2)	0.068(2)	0.098(2)	-0.014(1)	-0.003(2)	0.009(1)
C(17)	0.059(2)	0.080(2)	0.087(2)	-0.004(1)	0.017(1)	0.008(1)

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#

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;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

_publ_contact_letter

;The crystal structure of (+-)-(2R)-4-hydroxy-3-phenyl-
 2-benzylbutanenitrile (12c) is reported as supporting
 information in the article entitled, Hydroxy
 Alkenenitriles - Diastereoselective Conjugate
 Addition-Alkylations, which has been submitted
 to the Journal of Organic Chemistry for publication.
 The asymmetric unit consists of a single molecule.
 An intermolecular hydrogen bond is observed between
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;Fraser F. Fleming
;
;Department of Chemistry and Biochemistry
Duquesne University
Pittsburgh, PA 15282 USA
;
;Qunzhao Wang
;
;Department of Chemistry and Biochemistry
Duquesne University
Pittsburgh, PA 15282 USA
;
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;
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Duquesne University
Pittsburgh, PA 15282 USA
;

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Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations
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stereoselectively generates  $\beta$ -substituted hydroxynitriles.
t-BuMgCl-initiated deprotonation and chloride-alkyl
exchange from a second Grignard reagent, generates an
alkylmagnesium alkoxide that triggers conjugate and
generates a metallated nitrile. Sequential conjugate
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; ;The crystal structure of (+-)-(2R)-4-hydroxy-3-phenyl-
2-benzylbutanenitrile (12c) is reported as supporting
information in the article entitled, Hydroxy Alkenenitriles -
Diastereoselective Conjugate Addition-Alkylations,
which has been submitted to the Journal of the American

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Society for publication. The asymmetric unit consists of a single molecule. An intermolecular hydrogen bond is observed between the hydroxyl and nitrile groups:
O(1)-N(2) 2.865(2)\%A.

;

_publ_section_references

;

ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968).
Acta Cryst. A24, 351-359.

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The compound,(+)-(2R)-4-hydroxy-3-phenyl-2
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\y scans (North,Phillips & Matthews, 1968)
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(up to 4 scans to achieve I\s(I) > 3).
Stationary background counts were recorded at
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;Chelation-controlled conjugation addition of Grignard
reagents to cyclic and acyclic α -hydroxyalkenenitriles
stereoselectively generates β -substituted hydroxynitriles.
t-BuMgCl-initiated deprotonation and chloride-alkyl
exchange from a second Grignard reagent, generates an
alkylmagnesium alkoxide that triggers conjugate and
generates a metallated nitrile. Sequential conjugate
addition-alkylation with alkyl halide and carbonyl
electrophiles efficiently installs two new bonds and
up to three stereocenters in a single synthetic operation.
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; The crystal structure of (+-)-(3S,1R)-3-cyano-2-phenyl-3-(phenylcarbonyl)cyclohexyl benzoate (12d) is reported as supporting information in the article entitled, Hydroxy Alkenenitriles - Diastereoselective Conjugate Addition-Alkylations, which has been submitted to the Journal of Organic Chemistry for publication. The asymmetric unit consists of a single molecule.

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ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968).
Acta Cryst. A24, 351-359.

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\y scans (North,Phillips & Matthews, 1968)
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The scan width was (1.00+0.35tan\q)\% with an \w

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scan speed of 16.0 0\% per minute
 (up to 4 scans to achieve $I/\sigma(I) > 3$).
 Stationary background counts were recorded at each end of the
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C(10) 0.8405(2) 0.12624(8) -0.0873(1) 0.0438(5) 1.000 . Uani d ?

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C(11) 0.8111(2) 0.1464(1) -0.2021(2) 0.0569(6) 1.000 . Uani d ?
 C(12) 0.6803(3) 0.1349(1) -0.2694(2) 0.0738(7) 1.000 . Uani d ?
 C(13) 0.5779(2) 0.1020(1) -0.2226(2) 0.0732(7) 1.000 . Uani d ?
 C(14) 0.6076(2) 0.0794(1) -0.1092(2) 0.0565(6) 1.000 . Uani d ?
 C(15) 0.7474(2) 0.10212(9) 0.3382(1) 0.0404(5) 1.000 . Uani d ?
 C(16) 0.7408(2) 0.1731(1) 0.3600(2) 0.0523(6) 1.000 . Uani d ?
 C(17) 0.6294(2) 0.2013(1) 0.4018(2) 0.0641(7) 1.000 . Uani d ?
 C(18) 0.5236(2) 0.1591(1) 0.4230(2) 0.0679(7) 1.000 . Uani d ?
 C(19) 0.5285(2) 0.0887(1) 0.4032(2) 0.0677(7) 1.000 . Uani d ?
 C(20) 0.6393(2) 0.0604(1) 0.3609(2) 0.0530(6) 1.000 . Uani d ?
 C(21) 1.1366(2) 0.1567(1) 0.5121(1) 0.0473(5) 1.000 . Uani d ?
 C(22) 1.1699(2) 0.2710(1) 0.4167(2) 0.0490(5) 1.000 . Uani d ?
 C(23) 1.2303(2) 0.3365(1) 0.4211(2) 0.0606(6) 1.000 . Uani d ?
 C(24) 1.3189(2) 0.3590(1) 0.5211(2) 0.0680(7) 1.000 . Uani d ?
 C(25) 1.3483(2) 0.3160(1) 0.6162(2) 0.0702(7) 1.000 . Uani d ?
 C(26) 1.2874(2) 0.2508(1) 0.6134(2) 0.0585(6) 1.000 . Uani d ?
 C(27) 1.1972(2) 0.22791(9) 0.5131(1) 0.0430(5) 1.000 . Uani d ?
 H(1) 0.9927 -0.0053 0.1416 0.055 1.000 . Uiso c ?
 H(2) 1.0412 0.0581 0.0778 0.055 1.000 . Uiso c ?
 H(3) 1.2253 0.0210 0.2254 0.061 1.000 . Uiso c ?
 H(4) 1.1821 0.0964 0.2510 0.061 1.000 . Uiso c ?
 H(5) 1.2059 0.0301 0.4202 0.063 1.000 . Uiso c ?
 H(6) 1.0961 -0.0229 0.3566 0.063 1.000 . Uiso c ?
 H(7) 0.9839 0.0523 0.4646 0.048 1.000 . Uiso c ?
 H(8) 0.8453 0.0184 0.2934 0.047 1.000 . Uiso c ?
 H(9) 0.9313 0.1358 -0.0399 0.052 1.000 . Uiso c ?
 H(10) 0.8839 0.1679 -0.2367 0.068 1.000 . Uiso c ?
 H(11) 0.6576 0.1504 -0.3480 0.089 1.000 . Uiso c ?
 H(12) 0.4853 0.0952 -0.2697 0.086 1.000 . Uiso c ?
 H(13) 0.5362 0.0546 -0.0788 0.068 1.000 . Uiso c ?
 H(14) 0.8148 0.2025 0.3440 0.061 1.000 . Uiso c ?
 H(15) 0.6269 0.2510 0.4167 0.078 1.000 . Uiso c ?
 H(16) 0.4454 0.1777 0.4511 0.081 1.000 . Uiso c ?
 H(17) 0.4547 0.0590 0.4187 0.077 1.000 . Uiso c ?
 H(18) 0.6422 0.0115 0.3469 0.065 1.000 . Uiso c ?
 H(19) 1.1077 0.2553 0.3474 0.059 1.000 . Uiso c ?
 H(20) 1.2110 0.3664 0.3530 0.072 1.000 . Uiso c ?
 H(21) 1.3593 0.4057 0.5233 0.084 1.000 . Uiso c ?
 H(22) 1.4125 0.3311 0.6865 0.083 1.000 . Uiso c ?
 H(23) 1.3082 0.2199 0.6794 0.071 1.000 . Uiso c ?

loop_

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_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_12

_atom_site_aniso_U_13

_atom_site_aniso_U_23

O(1) 0.084(1) 0.082(1) 0.0577(9) -0.0504(9) 0.0180(8) -0.0072(7)

O(2) 0.0476(7) 0.0393(6) 0.0393(6) 0.0004(5) 0.0069(5) 0.0036(5)

O(3)	0.105(1)	0.0647(9)	0.0451(8)	-0.0014(8)	-0.0027(8)	0.0137(7)
N(1)	0.062(1)	0.0421(8)	0.0489(9)	-0.0088(7)	0.0157(7)	-0.0015(7)
C(1)	0.0406(9)	0.0326(8)	0.0402(9)	-0.0040(7)	0.0137(7)	-0.0017(6)
C(2)	0.055(1)	0.0412(9)	0.046(1)	0.0053(8)	0.0203(8)	-0.0010(8)
C(3)	0.048(1)	0.052(1)	0.053(1)	0.0117(8)	0.0200(9)	0.0039(8)
C(4)	0.054(1)	0.046(1)	0.052(1)	0.0124(8)	0.0156(9)	0.0077(8)
C(5)	0.051(1)	0.0340(9)	0.0406(9)	0.0033(7)	0.0152(8)	0.0073(7)
C(6)	0.0460(9)	0.0304(8)	0.0451(9)	-0.0038(7)	0.0178(8)	0.0021(7)
C(7)	0.0384(9)	0.0384(9)	0.0355(9)	-0.0041(7)	0.0117(7)	-0.0008(7)
C(8)	0.048(1)	0.0402(9)	0.048(1)	-0.0093(8)	0.0165(8)	-0.0074(8)
C(9)	0.0426(9)	0.0349(9)	0.046(1)	-0.0012(7)	0.0078(8)	-0.0071(7)
C(10)	0.048(1)	0.0392(9)	0.0437(9)	-0.0026(8)	0.0082(8)	-0.0016(7)
C(11)	0.077(1)	0.046(1)	0.046(1)	-0.007(1)	0.009(1)	0.0022(8)
C(12)	0.103(2)	0.055(1)	0.051(1)	-0.006(1)	-0.013(1)	0.007(1)
C(13)	0.070(1)	0.058(1)	0.075(2)	-0.002(1)	-0.024(1)	-0.003(1)
C(14)	0.047(1)	0.047(1)	0.072(1)	-0.0034(8)	0.003(1)	-0.0081(9)
C(15)	0.0397(9)	0.047(1)	0.0364(9)	-0.0013(7)	0.0117(7)	0.0035(7)
C(16)	0.055(1)	0.048(1)	0.061(1)	0.0040(9)	0.0265(9)	-0.0009(9)
C(17)	0.063(1)	0.072(1)	0.062(1)	0.020(1)	0.023(1)	-0.003(1)
C(18)	0.044(1)	0.113(2)	0.049(1)	0.022(1)	0.0148(9)	0.007(1)
C(19)	0.037(1)	0.107(2)	0.061(1)	-0.010(1)	0.0140(9)	0.010(1)
C(20)	0.047(1)	0.062(1)	0.051(1)	-0.0096(9)	0.0123(9)	0.0029(9)
C(21)	0.051(1)	0.053(1)	0.037(1)	0.0091(9)	0.0082(8)	0.0005(8)
C(22)	0.045(1)	0.052(1)	0.049(1)	-0.0006(8)	0.0068(8)	-0.0022(8)
C(23)	0.064(1)	0.055(1)	0.063(1)	-0.006(1)	0.014(1)	0.000(1)
C(24)	0.067(1)	0.066(1)	0.074(2)	-0.016(1)	0.021(1)	-0.018(1)
C(25)	0.065(1)	0.083(2)	0.060(1)	-0.012(1)	0.006(1)	-0.026(1)
C(26)	0.059(1)	0.074(1)	0.041(1)	0.004(1)	0.0064(9)	-0.0096(9)
C(27)	0.0378(9)	0.050(1)	0.0421(9)	0.0064(8)	0.0097(7)	-0.0054(8)

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O(1) C(8) 1.212(2) . . yes
O(2) C(5) 1.455(2) . . yes
O(2) C(21) 1.347(2) . . yes
O(3) C(21) 1.207(2) . . yes
N(1) C(7) 1.141(2) . . yes
C(1) C(2) 1.562(2) . . yes
C(1) C(6) 1.567(2) . . yes
C(1) C(7) 1.473(2) . . yes
C(1) C(8) 1.556(2) . . yes
C(2) C(3) 1.513(2) . . yes
C(2) H(1) 0.96 . . no
C(2) H(2) 0.96 . . no
C(3) C(4) 1.517(2) . . yes
C(3) H(3) 0.96 . . no
C(3) H(4) 0.96 . . no
C(4) C(5) 1.519(2) . . yes
C(4) H(5) 0.97 . . no
C(4) H(6) 0.96 . . no
C(5) C(6) 1.530(2) . . yes
C(5) H(7) 0.97 . . no
C(6) C(15) 1.519(2) . . yes
C(6) H(8) 0.96 . . no
C(8) C(9) 1.490(2) . . yes
C(9) C(10) 1.397(2) . . yes
C(9) C(14) 1.399(2) . . yes
C(10) C(11) 1.379(2) . . yes
C(10) H(9) 0.96 . . no
C(11) C(12) 1.372(3) . . yes
C(11) H(10) 0.98 . . no
C(12) C(13) 1.383(3) . . yes
C(12) H(11) 0.95 . . no
C(13) C(14) 1.377(3) . . yes
C(13) H(12) 0.97 . . no

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C(14) H(13) 0.97 . . no
 C(15) C(16) 1.389(2) . . yes
 C(15) C(20) 1.388(2) . . yes
 C(16) C(17) 1.387(2) . . yes
 C(16) H(14) 0.96 . . no
 C(17) C(18) 1.370(3) . . yes
 C(17) H(15) 0.97 . . no
 C(18) C(19) 1.371(3) . . yes
 C(18) H(16) 0.96 . . no
 C(19) C(20) 1.385(3) . . yes
 C(19) H(17) 0.96 . . no
 C(20) H(18) 0.95 . . no
 C(21) C(27) 1.485(2) . . yes
 C(22) C(23) 1.382(3) . . yes
 C(22) C(27) 1.385(2) . . yes
 C(22) H(19) 0.96 . . no
 C(23) C(24) 1.379(3) . . yes
 C(23) H(20) 0.97 . . no
 C(24) C(25) 1.373(3) . . yes
 C(24) H(21) 0.97 . . no
 C(25) C(26) 1.380(3) . . yes
 C(25) H(22) 0.97 . . no
 C(26) C(27) 1.391(2) . . yes
 C(26) H(23) 0.96 . . no
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 C(5) O(2) C(21) 116.5(1) . . . yes
 C(2) C(1) C(6) 108.6(1) . . . yes
 C(2) C(1) C(7) 110.2(1) . . . yes
 C(2) C(1) C(8) 105.9(1) . . . yes
 C(6) C(1) C(7) 111.7(1) . . . yes
 C(6) C(1) C(8) 109.9(1) . . . yes
 C(7) C(1) C(8) 110.3(1) . . . yes
 C(1) C(2) C(3) 113.3(1) . . . yes
 C(1) C(2) H(1) 108.8 . . . no
 C(1) C(2) H(2) 108.9 . . . no
 C(3) C(2) H(1) 108.8 . . . no
 C(3) C(2) H(2) 109.5 . . . no
 H(1) C(2) H(2) 107.5 . . . no
 C(2) C(3) C(4) 110.8(1) . . . yes
 C(2) C(3) H(3) 110.3 . . . no
 C(2) C(3) H(4) 109.2 . . . no
 C(4) C(3) H(3) 109.7 . . . no

C(4) C(3) H(4) 108.8 . . . no
 H(3) C(3) H(4) 108.0 . . . no
 C(3) C(4) C(5) 110.3(1) . . . yes
 C(3) C(4) H(5) 109.9 . . . no
 C(3) C(4) H(6) 109.6 . . . no
 C(5) C(4) H(5) 109.2 . . . no
 C(5) C(4) H(6) 110.5 . . . no
 H(5) C(4) H(6) 107.3 . . . no
 O(2) C(5) C(4) 108.7(1) . . . yes
 O(2) C(5) C(6) 109.2(1) . . . yes
 O(2) C(5) H(7) 108.9 . . . no
 C(4) C(5) C(6) 112.2(1) . . . yes
 C(4) C(5) H(7) 109.4 . . . no
 C(6) C(5) H(7) 108.3 . . . no
 C(1) C(6) C(5) 113.2(1) . . . yes
 C(1) C(6) C(15) 116.6(1) . . . yes
 C(1) C(6) H(8) 104.3 . . . no
 C(5) C(6) C(15) 112.5(1) . . . yes
 C(5) C(6) H(8) 104.3 . . . no
 C(15) C(6) H(8) 104.3 . . . no
 N(1) C(7) C(1) 178.2(2) . . . yes
 O(1) C(8) C(1) 117.8(2) . . . yes
 O(1) C(8) C(9) 120.1(2) . . . yes
 C(1) C(8) C(9) 122.0(1) . . . yes
 C(8) C(9) C(10) 124.5(1) . . . yes
 C(8) C(9) C(14) 117.2(2) . . . yes
 C(10) C(9) C(14) 118.2(2) . . . yes
 C(9) C(10) C(11) 120.7(2) . . . yes
 C(9) C(10) H(9) 119.4 . . . no
 C(11) C(10) H(9) 119.9 . . . no
 C(10) C(11) C(12) 120.3(2) . . . yes
 C(10) C(11) H(10) 120.2 . . . no
 C(12) C(11) H(10) 119.5 . . . no
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 C(11) C(12) H(11) 121.0 . . . no
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 C(12) C(13) C(14) 120.3(2) . . . yes
 C(12) C(13) H(12) 119.6 . . . no
 C(14) C(13) H(12) 120.1 . . . no
 C(9) C(14) C(13) 120.5(2) . . . yes
 C(9) C(14) H(13) 120.2 . . . no
 C(13) C(14) H(13) 119.3 . . . no
 C(6) C(15) C(16) 123.8(1) . . . yes
 C(6) C(15) C(20) 118.4(2) . . . yes
 C(16) C(15) C(20) 117.6(2) . . . yes
 C(15) C(16) C(17) 121.0(2) . . . yes
 C(15) C(16) H(14) 118.3 . . . no
 C(17) C(16) H(14) 120.7 . . . no
 C(16) C(17) C(18) 120.3(2) . . . yes
 C(16) C(17) H(15) 119.7 . . . no
 C(18) C(17) H(15) 120.0 . . . no

C(17) C(18) C(19) 119.8(2) . . . yes
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 C(19) C(18) H(16) 118.9 . . . no
 C(18) C(19) C(20) 120.2(2) . . . yes
 C(18) C(19) H(17) 119.8 . . . no
 C(20) C(19) H(17) 120.0 . . . no
 C(15) C(20) C(19) 121.2(2) . . . yes
 C(15) C(20) H(18) 118.9 . . . no
 C(19) C(20) H(18) 120.0 . . . no
 O(2) C(21) O(3) 123.3(2) . . . yes
 O(2) C(21) C(27) 111.9(1) . . . yes
 O(3) C(21) C(27) 124.8(2) . . . yes
 C(23) C(22) C(27) 120.1(2) . . . yes
 C(23) C(22) H(19) 120.4 . . . no
 C(27) C(22) H(19) 119.5 . . . no
 C(22) C(23) C(24) 120.2(2) . . . yes
 C(22) C(23) H(20) 119.7 . . . no
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 C(23) C(24) C(25) 119.9(2) . . . yes
 C(23) C(24) H(21) 119.2 . . . no
 C(25) C(24) H(21) 120.9 . . . no
 C(24) C(25) C(26) 120.5(2) . . . yes
 C(24) C(25) H(22) 120.3 . . . no
 C(26) C(25) H(22) 119.1 . . . no
 C(25) C(26) C(27) 119.8(2) . . . yes
 C(25) C(26) H(23) 121.2 . . . no
 C(27) C(26) H(23) 118.9 . . . no
 C(21) C(27) C(22) 122.1(2) . . . yes
 C(21) C(27) C(26) 118.4(2) . . . yes
 C(22) C(27) C(26) 119.4(2) . . . yes

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 O(1) C(24) 3.454(3) . 2_745 no
 N(1) C(27) 3.406(2) . 4_554 no
 N(1) C(11) 3.450(2) . 4 no
 N(1) C(26) 3.527(3) . 4_554 no

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 O(1) C(8) C(1) C(7) -143.5(2) no
 O(1) C(8) C(9) C(10) -161.1(2) no
 O(1) C(8) C(9) C(14) 15.9(2) no
 O(2) C(5) C(4) C(3) -64.3(2) no
 O(2) C(5) C(6) C(1) 67.1(2) no
 O(2) C(5) C(6) C(15) -67.8(2) no
 O(2) C(21) C(27) C(22) -0.6(2) no
 O(2) C(21) C(27) C(26) -178.8(2) no
 O(3) C(21) O(2) C(5) -7.3(2) no
 O(3) C(21) C(27) C(22) 178.0(2) no
 O(3) C(21) C(27) C(26) -0.1(3) no
 N(1) C(7) C(1) C(2) 72(6) no
 N(1) C(7) C(1) C(6) -167(6) no
 N(1) C(7) C(1) C(8) -44(6) no
 C(1) C(2) C(3) C(4) 58.1(2) no
 C(1) C(6) C(5) C(4) -53.5(2) no
 C(1) C(6) C(15) C(16) -66.8(2) no
 C(1) C(6) C(15) C(20) 117.6(2) no
 C(1) C(8) C(9) C(10) 14.7(2) no
 C(1) C(8) C(9) C(14) -168.2(2) no
 C(2) C(1) C(6) C(5) 49.6(2) no
 C(2) C(1) C(6) C(15) -177.5(1) no
 C(2) C(1) C(8) C(9) -78.8(2) no
 C(2) C(3) C(4) C(5) -58.5(2) no
 C(3) C(2) C(1) C(6) -52.3(2) no
 C(3) C(2) C(1) C(7) 70.3(2) no
 C(3) C(2) C(1) C(8) -170.3(1) no
 C(3) C(4) C(5) C(6) 56.6(2) no
 C(4) C(5) O(2) C(21) -80.9(2) no
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 C(5) O(2) C(21) C(27) 171.4(1) no
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;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

_publ_contact_letter

;The crystal structure of 5-(hydroxyphenylmethyl)-7-oxo-8-phenylbicyclo[3.2.1]octan-6-one (12f) is reported as supporting information in the article entitled, Hydroxy Alkenenitriles - Diastereoselective Conjugate Addition-Alkylations, which has been submitted to the Journal of Organic Chemistry for publication. The asymmetric unit consists of a single molecule. An intermolecular hydrogen bond is observed between the hydroxyl group and carbonyl oxygen of the lactone: O(2)-O(3) 2.87(1)\%A.

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;Fraser F. Fleming
;
;Department of Chemistry and Biochemistry
Duquesne University
Pittsburgh, PA 15282 USA
;
;Qunzhao Wang
;
;Department of Chemistry and Biochemistry
Duquesne University
Pittsburgh, PA 15282 USA
;
;Omar W.. Steward
;
;Department of Chemistry and Biochemistry
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Pittsburgh, PA 15282 USA
;

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Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations
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;Chelation-controlled conjugation addition of Grignard
reagents to cyclic and acyclic α -hydroxyalkenenitriles
stereoselectively generates β -substituted hydroxynitriles.
t-BuMgCl-initiated deprotonation and chloride-alkyl
exchange from a second Grignard reagent, generates an
alkylmagnesium alkoxide that triggers conjugate and
generates a metallated nitrile. Sequential conjugate
addition-alkylation with alkyl halide and carbonyl
electrophiles efficiently installs two new bonds and
up to three stereocenters in a single synthetic operation.
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;

ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan. Single Crystal Structure Analysis Software. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968). Acta Cryst. A24, 351-359.

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The 5-(hydroxyphenylmethyl)-7-oxo-8-phenyl-bicyclo-
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compound 12f was crystallized from 1:2
EtOAc/hexanes, m.p. 413.2-414.7 K.

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Stationary background counts were recorded at each end of the
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C(5) 0.093(1) -0.0282(9) 0.4050(8) 0.058(5) 1.000 . Uani d ?
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C(15) 0.170(1) 0.110(1) 0.1481(8) 0.046(4) 1.000 . Uani d ?
C(16) 0.168(1) 0.056(1) 0.068(1) 0.065(5) 1.000 . Uani d ?
C(17) 0.170(2) 0.111(2) -0.011(1) 0.085(6) 1.000 . Uani d ?
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C(3) 0.052(9) 0.042(8) 0.057(8) -0.015(7) 0.003(7) -0.018(8)
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C(5) 0.07(1) 0.047(8) 0.08(1) -0.007(7) 0.044(8) 0.000(7)
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C(3) C(4) 1.50(1) . . yes
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;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

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;The crystal structure of (+-)-(1R,4R)-3-cyano-

1-methyl-4-(4-nitrophenylcarbonyloxy)-2,4-diphenylbutyl

4-nitrobenzoate (13a di-4-nitrobenzoate ester) is reported

as supporting information in the article entitled,

Hydroxy Alkenenitriles - Diastereoselective Conjugate

Addition-Alkylations, which has been submitted to the

Journal of Organic Chemistry for publication.

The crystal were found to have a methylene chloride

of crystallization. The asymmetric unit consists of

13a di-4-nitrobenzoate ester and a methylene chloride

of crystallization.

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;Fraser F. Fleming

;

;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

;Qunzhao Wang

;

;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

;

;Omar W.. Steward

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;Department of Chemistry and Biochemistry

Duquesne University

Pittsburgh, PA 15282 USA

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Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations
;

_publ_section_title_footnote 'ENTER ANY FOOTNOTES TO TITLE'

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stereoselectively generates  $\beta$ -substituted hydroxynitriles.
t-BuMgCl-initiated deprotonation and chloride-alkyl
exchange from a second Grignard reagent, generates an
alkylmagnesium alkoxide that triggers conjugate and
generates a metallated nitrile. Sequential conjugate
addition-alkylation with alkyl halide and carbonyl
electrophiles efficiently installs two new bonds and
up to three stereocenters in a single synthetic operation.
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1-methyl-4-(4-nitrophenylcarbonyloxy)-2,4-diphenylbutyl
4-nitrobenzoate (13a di-4-nitrobenzoate ester) is reported
as supporting information in the article entitled,
Hydroxy Alkenenitriles - Diastereoselective Conjugate
Addition-Alkylations, which has been submitted to the
Journal of Organic Chemistry for publication.
The crystal were found to have a methylene chloride
of crystallization. The asymmetric unit consists of
13a di-4-nitrobenzoate ester and a methylene chloride
of crystallization.
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ENTER OTHER REFERENCES

Molecular Structure Corporation. (1992-1997). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

North, A.C.T., Phillips, D. C. & Mathews, F. S. (1968).
Acta Cryst. A24, 351-359.

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1-methyl-4-(4-nitrophenylcarbonyloxy)-
 2,4-diphenylbutyl 4-nitrobenzoate
 (13a di-4-nitrobenzoate ester) was
 synthesized as described in the paper.
 The compound (13a di-4-nitrobenzoate ester)
 was crystallized from 1:2 EtOAc/hexanes,
 followed by recrystallization from
 EtOAc/hexanes/methylene chloride.
 The crystals were found to contain a
 methylene chloride of crystallization
 by X-ray diffraction. The crystals
 were observed to have two melting points:
 m.p. 409-414 K forming a white powder
 (probably resulting from loss of the
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 m.p. 463-465 K.
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;\y scans (North,Phillips & Matthews, 1968)

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Stationary background counts were recorded at each end of the
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C(6) 0.580(1) 0.116(1) 0.0594(4) 0.048(4) 1.000 . Uani d ?

```


C(7) 0.6018(9) 0.047(1) 0.1732(4) 0.044(4) 1.000 . Uani d ?
 C(8) 0.654(1) 0.124(1) 0.2091(5) 0.064(4) 1.000 . Uani d ?
 C(9) 0.600(1) 0.197(1) 0.2486(6) 0.089(6) 1.000 . Uani d ?
 C(10) 0.498(1) 0.188(1) 0.2537(5) 0.076(5) 1.000 . Uani d ?
 C(11) 0.444(1) 0.112(1) 0.2178(6) 0.074(5) 1.000 . Uani d ?
 C(12) 0.495(1) 0.040(1) 0.1780(4) 0.057(4) 1.000 . Uani d ?
 C(13) 0.7546(8) 0.083(1) -0.0143(4) 0.043(3) 1.000 . Uani d ?
 C(14) 0.8000(9) 0.207(1) -0.0050(4) 0.059(4) 1.000 . Uani d ?
 C(15) 0.822(1) 0.284(1) -0.0525(7) 0.086(5) 1.000 . Uani d ?
 C(16) 0.799(1) 0.238(2) -0.1046(5) 0.086(6) 1.000 . Uani d ?
 C(17) 0.754(1) 0.122(1) -0.1118(5) 0.078(5) 1.000 . Uani d ?
 C(18) 0.732(1) 0.046(1) -0.0664(5) 0.064(4) 1.000 . Uani d ?
 C(19) 0.609(1) -0.247(1) 0.1408(4) 0.056(4) 1.000 . Uani d ?
 C(20) 0.5377(9) -0.348(1) 0.1207(4) 0.045(4) 1.000 . Uani d ?
 C(21) 0.485(1) -0.335(1) 0.0719(4) 0.055(4) 1.000 . Uani d ?
 C(22) 0.423(1) -0.430(1) 0.0542(5) 0.074(5) 1.000 . Uani d ?
 C(23) 0.411(1) -0.545(1) 0.0884(6) 0.066(4) 1.000 . Uani d ?
 C(24) 0.464(1) -0.555(1) 0.1345(6) 0.083(5) 1.000 . Uani d ?
 C(25) 0.527(1) -0.461(1) 0.1521(5) 0.069(4) 1.000 . Uani d ?
 C(26) 0.944(1) -0.010(1) 0.1256(5) 0.056(4) 1.000 . Uani d ?
 C(27) 1.0095(9) 0.093(1) 0.1492(4) 0.050(4) 1.000 . Uani d ?
 C(28) 1.020(1) 0.215(1) 0.1215(4) 0.053(4) 1.000 . Uani d ?
 C(29) 1.086(1) 0.307(1) 0.1434(4) 0.052(4) 1.000 . Uani d ?
 C(30) 1.1367(9) 0.278(1) 0.1908(4) 0.046(4) 1.000 . Uani d ?
 C(31) 1.132(1) 0.164(1) 0.2173(4) 0.059(4) 1.000 . Uani d ?
 C(32) 1.065(1) 0.071(1) 0.1974(4) 0.061(4) 1.000 . Uani d ?
 C(33) 0.679(2) 0.039(2) 0.392(2) 0.42(2) 1.000 . Uani d ?
 H(1) 0.7212 -0.0514 0.1448 0.049 1.000 . Uiso c ?
 H(2) 0.7185 0.1320 0.0914 0.050 1.000 . Uiso c ?
 H(3) 0.6932 -0.0758 0.0180 0.051 1.000 . Uiso c ?
 H(4) 0.8144 -0.1420 0.0786 0.045 1.000 . Uiso c ?
 H(5) 0.8542 -0.2004 -0.0065 0.074 1.000 . Uiso c ?
 H(6) 0.9124 -0.0702 -0.0139 0.074 1.000 . Uiso c ?
 H(7) 0.9517 -0.1730 0.0276 0.074 1.000 . Uiso c ?
 H(8) 0.7261 0.1293 0.2076 0.077 1.000 . Uiso c ?
 H(9) 0.6366 0.2541 0.2721 0.106 1.000 . Uiso c ?
 H(10) 0.4646 0.2338 0.2821 0.091 1.000 . Uiso c ?
 H(11) 0.3715 0.1079 0.2198 0.089 1.000 . Uiso c ?
 H(12) 0.4572 -0.0148 0.1538 0.069 1.000 . Uiso c ?
 H(13) 0.8152 0.2372 0.0308 0.071 1.000 . Uiso c ?
 H(14) 0.8519 0.3676 -0.0484 0.103 1.000 . Uiso c ?
 H(15) 0.8157 0.2888 -0.1355 0.102 1.000 . Uiso c ?
 H(16) 0.7376 0.0929 -0.1475 0.093 1.000 . Uiso c ?
 H(17) 0.6987 -0.0359 -0.0721 0.077 1.000 . Uiso c ?
 H(18) 0.4933 -0.2584 0.0507 0.066 1.000 . Uiso c ?
 H(19) 0.3869 -0.4227 0.0204 0.089 1.000 . Uiso c ?
 H(20) 0.4569 -0.6316 0.1558 0.099 1.000 . Uiso c ?
 H(21) 0.5638 -0.4706 0.1854 0.083 1.000 . Uiso c ?
 H(22) 0.9829 0.2321 0.0887 0.063 1.000 . Uiso c ?
 H(23) 1.0954 0.3891 0.1259 0.063 1.000 . Uiso c ?
 H(24) 1.1721 0.1480 0.2489 0.071 1.000 . Uiso c ?

H(25) 1.0571 -0.0092 0.2165 0.073 1.000 . Uiso c ?
H(26) 0.6609 0.1080 0.3689 0.505 1.000 . Uiso c ?
H(27) 0.7395 0.0625 0.4124 0.505 1.000 . Uiso c ?

loop_

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_atom_site_aniso_U_11

_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_12

_atom_site_aniso_U_13

_atom_site_aniso_U_23

Cl(1)	0.265(9)	0.127(4)	0.224(6)	0.024(6)	-0.087(6)	-0.052(4)
Cl(2)	0.218(8)	0.157(6)	0.42(1)	-0.007(6)	0.137(8)	0.051(7)
O(1)	0.048(5)	0.038(4)	0.050(4)	-0.018(5)	0.003(4)	-0.001(4)
O(2)	0.121(9)	0.066(6)	0.089(6)	-0.018(6)	-0.047(7)	0.016(5)
O(3)	0.11(1)	0.103(9)	0.19(1)	-0.022(8)	-0.08(1)	-0.001(9)
O(4)	0.083(8)	0.060(6)	0.20(1)	-0.025(7)	0.053(8)	-0.030(7)
O(5)	0.042(5)	0.046(4)	0.048(4)	-0.007(4)	-0.024(4)	0.012(4)
O(6)	0.096(8)	0.086(7)	0.137(8)	-0.048(7)	-0.062(7)	0.054(6)
O(7)	0.084(8)	0.073(7)	0.113(7)	-0.011(7)	-0.025(6)	-0.022(6)
O(8)	0.111(8)	0.119(8)	0.064(5)	-0.017(7)	-0.053(6)	-0.022(5)
N(1)	0.075(9)	0.094(9)	0.077(7)	0.031(8)	0.007(7)	0.023(6)
N(2)	0.06(1)	0.036(8)	0.20(2)	-0.008(8)	0.03(1)	-0.01(1)
N(3)	0.064(8)	0.056(7)	0.063(7)	0.002(8)	0.003(7)	-0.013(7)
C(1)	0.036(7)	0.038(6)	0.050(6)	-0.006(7)	0.004(6)	-0.005(5)
C(2)	0.049(8)	0.032(6)	0.044(6)	-0.008(7)	0.005(6)	-0.001(5)
C(3)	0.036(7)	0.044(7)	0.047(6)	-0.007(6)	0.007(6)	-0.004(6)
C(4)	0.041(8)	0.033(6)	0.039(5)	-0.003(7)	0.014(6)	-0.005(5)
C(5)	0.059(9)	0.062(8)	0.065(7)	0.000(8)	0.003(7)	-0.003(6)
C(6)	0.046(9)	0.052(8)	0.046(7)	-0.002(8)	0.006(7)	0.010(6)
C(7)	0.040(8)	0.051(8)	0.041(6)	-0.014(7)	-0.002(6)	0.003(6)
C(8)	0.062(9)	0.068(9)	0.064(7)	-0.011(8)	-0.002(8)	-0.021(7)
C(9)	0.10(1)	0.07(1)	0.09(1)	-0.01(1)	0.01(1)	-0.031(8)
C(10)	0.07(1)	0.09(1)	0.064(9)	0.01(1)	0.008(9)	-0.031(8)
C(11)	0.06(1)	0.066(9)	0.095(9)	0.012(9)	0.043(9)	0.014(8)
C(12)	0.07(1)	0.057(8)	0.041(6)	-0.007(9)	0.015(7)	-0.005(6)
C(13)	0.031(7)	0.055(7)	0.044(6)	-0.004(7)	0.003(6)	0.011(6)
C(14)	0.050(9)	0.053(8)	0.075(8)	0.005(8)	0.009(7)	0.029(7)
C(15)	0.06(1)	0.054(8)	0.14(1)	-0.003(8)	-0.01(1)	0.032(9)
C(16)	0.08(1)	0.14(1)	0.046(8)	0.01(1)	0.016(8)	0.020(9)
C(17)	0.10(1)	0.08(1)	0.055(8)	-0.02(1)	0.018(9)	0.003(8)
C(18)	0.07(1)	0.055(7)	0.070(8)	-0.004(8)	-0.014(8)	-0.012(7)
C(19)	0.09(1)	0.046(8)	0.033(6)	-0.010(9)	0.011(8)	0.009(7)
C(20)	0.050(8)	0.035(7)	0.050(6)	-0.003(7)	0.005(7)	0.007(6)
C(21)	0.062(9)	0.041(7)	0.063(7)	-0.008(8)	0.003(7)	-0.015(6)
C(22)	0.07(1)	0.062(9)	0.094(9)	-0.003(9)	-0.011(8)	-0.008(8)
C(23)	0.040(8)	0.037(8)	0.12(1)	-0.012(8)	0.001(8)	0.003(8)
C(24)	0.09(1)	0.07(1)	0.092(9)	-0.02(1)	-0.02(1)	0.034(8)
C(25)	0.08(1)	0.044(8)	0.084(8)	-0.027(9)	0.012(8)	-0.012(7)
C(26)	0.052(9)	0.042(7)	0.075(8)	-0.002(8)	0.019(8)	0.017(7)

```

C(27) 0.039(8) 0.043(7) 0.068(7) -0.002(7) -0.017(7) -0.002(6)
C(28) 0.058(9) 0.049(8) 0.052(6) 0.004(8) -0.007(7) -0.002(6)
C(29) 0.060(9) 0.045(7) 0.051(7) -0.010(8) -0.003(7) 0.003(6)
C(30) 0.045(8) 0.050(8) 0.042(6) -0.009(7) 0.000(7) -0.009(6)
C(31) 0.058(9) 0.062(8) 0.056(7) -0.006(8) -0.024(7) 0.009(7)
C(32) 0.067(9) 0.074(9) 0.040(6) -0.015(9) -0.027(7) 0.034(6)
C(33) 0.20(3) 0.07(1) 1.0(1) -0.01(2) 0.36(5) 0.00(3)
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_refine_ls_wR_factor_all ?
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_refine_diff_density_max 0.36
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Cl(1) C(33) 1.61(3) . . yes
Cl(2) C(33) 1.76(3) . . yes
O(1) C(1) 1.46(1) . . yes
O(1) C(19) 1.34(1) . . yes

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O(2) C(19) 1.18(1) . . yes
O(3) N(2) 1.17(2) . . yes
O(4) N(2) 1.29(1) . . yes
O(5) C(4) 1.46(1) . . yes
O(5) C(26) 1.33(1) . . yes
O(6) C(26) 1.22(1) . . yes
O(7) N(3) 1.23(1) . . yes
O(8) N(3) 1.18(1) . . yes
N(1) C(6) 1.14(1) . . yes
N(2) C(23) 1.52(2) . . yes
N(3) C(30) 1.49(1) . . yes
C(1) C(2) 1.53(1) . . yes
C(1) C(7) 1.48(1) . . yes
C(1) H(1) 0.95 . . no
C(2) C(3) 1.59(1) . . yes
C(2) C(6) 1.48(2) . . yes
C(2) H(2) 0.95 . . no
C(3) C(4) 1.53(1) . . yes
C(3) C(13) 1.49(1) . . yes
C(3) H(3) 0.95 . . no
C(4) C(5) 1.42(1) . . yes
C(4) H(4) 0.95 . . no
C(5) H(5) 0.95 . . no
C(5) H(6) 0.95 . . no
C(5) H(7) 0.95 . . no
C(7) C(8) 1.37(1) . . yes
C(7) C(12) 1.42(2) . . yes
C(8) C(9) 1.42(2) . . yes
C(8) H(8) 0.95 . . no
C(9) C(10) 1.35(2) . . yes
C(9) H(9) 0.95 . . no
C(10) C(11) 1.38(2) . . yes
C(10) H(10) 0.95 . . no
C(11) C(12) 1.40(1) . . yes
C(11) H(11) 0.95 . . no
C(12) H(12) 0.95 . . no
C(13) C(14) 1.42(1) . . yes
C(13) C(18) 1.37(1) . . yes
C(14) C(15) 1.44(2) . . yes
C(14) H(13) 0.95 . . no
C(15) C(16) 1.39(2) . . yes
C(15) H(14) 0.95 . . no
C(16) C(17) 1.33(2) . . yes
C(16) H(15) 0.95 . . no
C(17) C(18) 1.40(2) . . yes
C(17) H(16) 0.95 . . no
C(18) H(17) 0.95 . . no
C(19) C(20) 1.48(1) . . yes
C(20) C(21) 1.38(1) . . yes
C(20) C(25) 1.40(1) . . yes
C(21) C(22) 1.35(1) . . yes

C(21) H(18) 0.95 . . no
 C(22) C(23) 1.45(2) . . yes
 C(22) H(19) 0.95 . . no
 C(23) C(24) 1.32(2) . . yes
 C(24) C(25) 1.34(2) . . yes
 C(24) H(20) 0.95 . . no
 C(25) H(21) 0.95 . . no
 C(26) C(27) 1.48(1) . . yes
 C(27) C(28) 1.43(1) . . yes
 C(27) C(32) 1.40(1) . . yes
 C(28) C(29) 1.39(1) . . yes
 C(28) H(22) 0.95 . . no
 C(29) C(30) 1.37(1) . . yes
 C(29) H(23) 0.95 . . no
 C(30) C(31) 1.34(1) . . yes
 C(31) C(32) 1.38(1) . . yes
 C(31) H(24) 0.95 . . no
 C(32) H(25) 0.95 . . no
 C(33) H(26) 0.94 . . no
 C(33) H(27) 0.96 . . no
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 C(1) O(1) C(19) 117.8(8) . . . yes
 C(4) O(5) C(26) 118.0(8) . . . yes
 O(3) N(2) O(4) 129(2) . . . yes
 O(3) N(2) C(23) 123(2) . . . yes
 O(4) N(2) C(23) 108(2) . . . yes
 O(7) N(3) O(8) 122(1) . . . yes
 O(7) N(3) C(30) 117(1) . . . yes
 O(8) N(3) C(30) 122(1) . . . yes
 O(1) C(1) C(2) 106.4(7) . . . yes
 O(1) C(1) C(7) 112.2(9) . . . yes
 O(1) C(1) H(1) 108.4 . . . no
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 C(2) C(1) H(1) 108.5 . . . no
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 C(1) C(2) C(3) 115.6(8) . . . yes
 C(1) C(2) C(6) 109.5(8) . . . yes
 C(1) C(2) H(2) 106.9 . . . no
 C(3) C(2) C(6) 110.6(8) . . . yes
 C(3) C(2) H(2) 106.9 . . . no
 C(6) C(2) H(2) 107.0 . . . no
 C(2) C(3) C(4) 111.3(8) . . . yes

C(2) C(3) C(13) 110.8(8) . . . yes
 C(2) C(3) H(3) 106.6 . . . no
 C(4) C(3) C(13) 114.2(8) . . . yes
 C(4) C(3) H(3) 106.7 . . . no
 C(13) C(3) H(3) 106.7 . . . no
 O(5) C(4) C(3) 106.3(7) . . . yes
 O(5) C(4) C(5) 109.0(9) . . . yes
 O(5) C(4) H(4) 109.1 . . . no
 C(3) C(4) C(5) 114.2(8) . . . yes
 C(3) C(4) H(4) 109.1 . . . no
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 N(1) C(6) C(2) 177(1) . . . yes
 C(1) C(7) C(8) 120(1) . . . yes
 C(1) C(7) C(12) 122(1) . . . yes
 C(8) C(7) C(12) 118(1) . . . yes
 C(7) C(8) C(9) 120(1) . . . yes
 C(7) C(8) H(8) 120.2 . . . no
 C(9) C(8) H(8) 120.3 . . . no
 C(8) C(9) C(10) 122(1) . . . yes
 C(8) C(9) H(9) 119.0 . . . no
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 C(12) C(11) H(11) 120.0 . . . no
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 C(3) C(13) C(14) 120.9(9) . . . yes
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 C(14) C(13) C(18) 119(1) . . . yes
 C(13) C(14) C(15) 117(1) . . . yes
 C(13) C(14) H(13) 121.7 . . . no
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 C(14) C(15) H(14) 119.8 . . . no
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 C(15) C(16) C(17) 121(1) . . . yes
 C(15) C(16) H(15) 119.6 . . . no
 C(17) C(16) H(15) 119.3 . . . no
 C(16) C(17) C(18) 119(1) . . . yes
 C(16) C(17) H(16) 120.3 . . . no
 C(18) C(17) H(16) 120.3 . . . no

C(13) C(18) C(17) 123(1) . . . yes
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 O(1) C(19) O(2) 123(1) . . . yes
 O(1) C(19) C(20) 112(1) . . . yes
 O(2) C(19) C(20) 126(1) . . . yes
 C(19) C(20) C(21) 122(1) . . . yes
 C(19) C(20) C(25) 118(1) . . . yes
 C(21) C(20) C(25) 120(1) . . . yes
 C(20) C(21) C(22) 121(1) . . . yes
 C(20) C(21) H(18) 119.6 . . . no
 C(22) C(21) H(18) 119.6 . . . no
 C(21) C(22) C(23) 118(1) . . . yes
 C(21) C(22) H(19) 121.2 . . . no
 C(23) C(22) H(19) 121.3 . . . no
 N(2) C(23) C(22) 113(1) . . . yes
 N(2) C(23) C(24) 127(1) . . . yes
 C(22) C(23) C(24) 120(1) . . . yes
 C(23) C(24) C(25) 122(1) . . . yes
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 C(25) C(24) H(20) 119.0 . . . no
 C(20) C(25) C(24) 119(1) . . . yes
 C(20) C(25) H(21) 120.6 . . . no
 C(24) C(25) H(21) 120.3 . . . no
 O(5) C(26) O(6) 122(1) . . . yes
 O(5) C(26) C(27) 115.1(9) . . . yes
 O(6) C(26) C(27) 122(1) . . . yes
 C(26) C(27) C(28) 120(1) . . . yes
 C(26) C(27) C(32) 121(1) . . . yes
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 C(27) C(28) C(29) 118(1) . . . yes
 C(27) C(28) H(22) 120.8 . . . no
 C(29) C(28) H(22) 120.7 . . . no
 C(28) C(29) C(30) 118(1) . . . yes
 C(28) C(29) H(23) 120.9 . . . no
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 C(29) C(30) C(31) 126(1) . . . yes
 C(30) C(31) C(32) 118(1) . . . yes
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 C(32) C(31) H(24) 121.3 . . . no
 C(27) C(32) C(31) 121(1) . . . yes
 C(27) C(32) H(25) 119.6 . . . no
 C(31) C(32) H(25) 119.7 . . . no
 Cl(1) C(33) Cl(2) 118(1) . . . yes
 Cl(1) C(33) H(26) 108.2 . . . no
 Cl(1) C(33) H(27) 106.8 . . . no
 Cl(2) C(33) H(26) 108.0 . . . no
 Cl(2) C(33) H(27) 106.6 . . . no
 H(26) C(33) H(27) 109.3 . . . no

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Cl(1)  C(28)   3.72(1)  . 2_645 no
Cl(1)  C(5)    3.77(1)  . 2_655 no
Cl(2)  C(28)   3.68(1)  . 2_645 no
Cl(2)  C(8)    3.69(1)  . 2_645 no
Cl(2)  C(25)   3.71(2)  . 2_655 no
O(2)   C(33)   3.48(3)  . 2_645 no
O(2)   C(9)    3.62(2)  . 2_645 no
O(2)   C(8)    3.84(2)  . 2_645 no
O(3)   N(1)    3.45(2)  . 1_545 no
O(3)   C(5)    3.58(2)  . 3_645 no
O(3)   C(21)   3.60(2)  . 3_645 no
O(3)   C(33)   3.67(5)  . 4_444 no
O(3)   C(4)    3.89(2)  . 3_645 no
O(3)   C(3)    3.89(2)  . 3_645 no
O(4)   N(1)    3.15(2)  . 1_545 no
O(4)   N(3)    3.22(1)  . 1_445 no
O(4)   C(30)   3.22(2)  . 1_445 no
O(4)   C(29)   3.22(2)  . 1_445 no
O(4)   O(7)    3.26(1)  . 1_445 no
O(4)   C(18)   3.45(1)  . 3_645 no
O(4)   C(11)   3.67(2)  . 1_545 no
O(4)   C(12)   3.82(2)  . 1_545 no
O(4)   O(8)    3.82(1)  . 1_445 no
O(5)   C(5)    3.80(1)  . 3_755 no
O(6)   C(9)    3.25(2)  . 2_645 no
O(6)   C(10)   3.27(2)  . 2_645 no
O(6)   C(16)   3.81(2)  . 3_755 no
O(7)   C(11)   3.43(2)  . 2_655 no
O(7)   C(24)   3.52(2)  . 1_665 no
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O(7)   C(31)   3.58(1)  . 2_755 no
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O(8)   C(32)   3.43(1)  . 2_755 no
O(8)   C(10)   3.53(2)  . 1_655 no
O(8)   C(11)   3.54(2)  . 1_655 no
O(8)   C(31)   3.58(2)  . 2_755 no
O(8)   C(16)   3.67(2)  . 4 no
O(8)   C(11)   3.88(2)  . 2_655 no

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N(1)	C(24)	3.79(2)	. 1_565	no
N(1)	C(22)	3.82(2)	. 3_655	no
N(1)	C(6)	3.88(1)	. 3_655	no
N(2)	C(29)	3.88(2)	. 1_445	no
N(3)	C(11)	3.62(2)	. 2_655	no
C(5)	C(28)	3.56(2)	. 3_755	no
C(15)	C(22)	3.55(2)	. 3_655	no
C(16)	C(22)	3.74(2)	. 3_655	no
C(17)	C(32)	3.75(2)	. 3_755	no
C(19)	C(33)	3.65(2)	. 2_645	no
C(22)	C(22)	3.65(3)	. 3_645	no

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loop_

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_geom_torsion_publ_flag

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O(1) C(1) C(2) C(6) 69(1) no

O(1) C(1) C(7) C(8) 156.2(9) no

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O(1) C(19) C(20) C(21) 10(2) no

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O(2) C(19) C(20) C(21) -175(1) no

O(2) C(19) C(20) C(25) 4(2) no

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O(3) N(2) C(23) C(24) -169(2) no

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 N(2) C(23) C(22) C(21) 179(1) no
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 C(1) O(1) C(19) C(20) 171.8(8) no
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