Organo-Catalytic Asymmetric Conjugate Addition of Nitroalkanes to α,β-Unsaturated Enones Using Novel Imidazoline Catalysts

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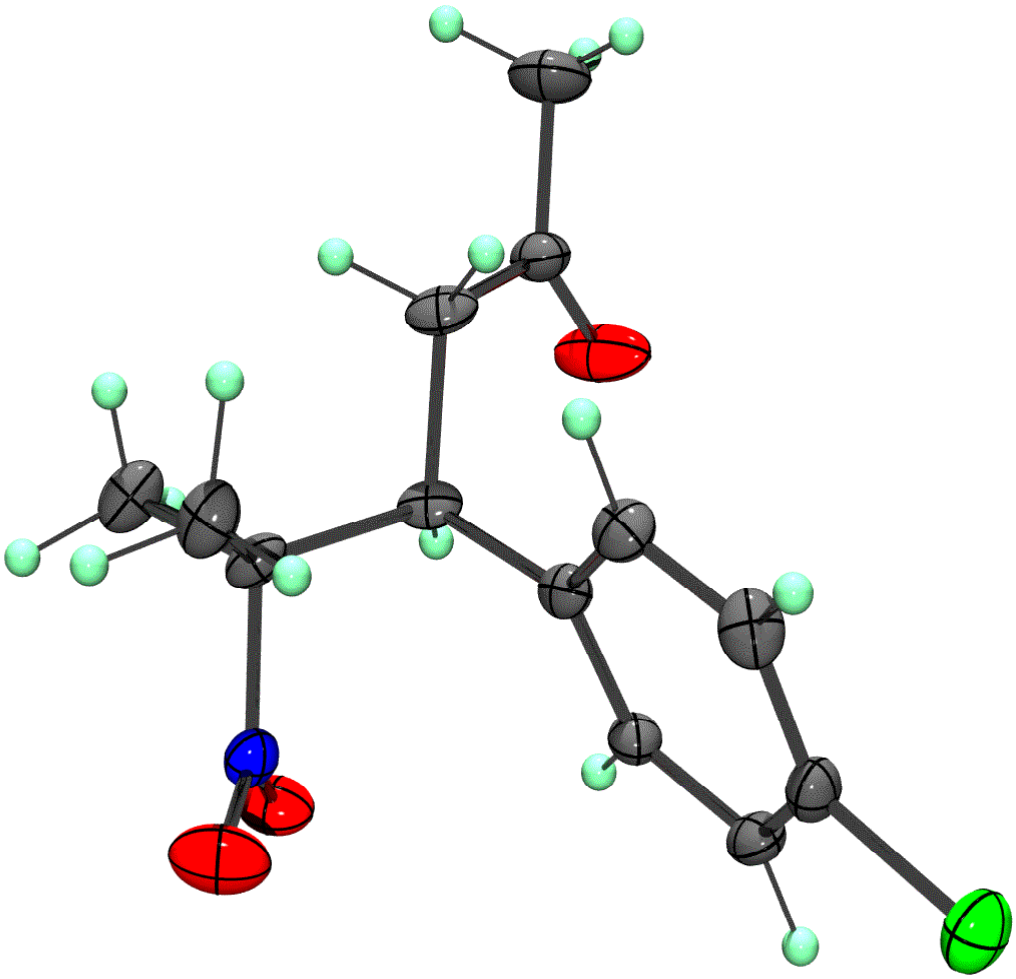
Supporting Information

Contents

X-Ray Structure of 4-(4-Chloro-Phenyl)-5-Methyl-5-Nitro-Hexan-2-One, 3j.



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      #     CCDC ELECTRONIC DATA DEPOSITION FORM (CIF, VERSION DEC95)


      #     This electronic data deposition form must be associated with CIF
      #     output and can be used :
      #     (a) WHEN SUBMITTING A PAPER TO A JOURNAL FOR PUBLICATION or
      #     (b) WHEN SUBMITTING A PRIVATE COMMUNICATION TO THE CCDC

      #     A comprehensive archive of CIF resources is maintained by the IUCr
      #     at its Chester office. This can be accessed
      #     by anonymous ftp to ftp.iucr.ac.uk
      #     or via the WWW at http://www.iucr.ac.uk/cif/home.html

      #     If data items in this form are not available or not applicable
      #     then ignore these items

      #     A few items specific to CCDC input are indicated by _ccdc_

      #     Submission and help information is provided at the end of this for

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      #1    Data block identification for start of deposition

data_cpd-3j

      #-------------------------------------------------------------------------
      #2    Person making the deposition

_publ_contact_author                'Rita Hazell'
_publ_contact_author_email           haz@chem.au.dk

      #-------------------------------------------------------------------------
      #3    Publication details

      #     Provide these details if the structure has been published,
      #     accepted or submitted for publication

      #     The CCDC journal deposition number, eg. 182/357,
      #     should be included only if it has been assigned by the journal

loop_
_publ_author_name
'Nis Halland'
'Rita Hazell'
'Karl Anker Jorgensen'

_journal_name_full                  'J. Org. Chem.'
_journal_volume                     '?'
_journal_page_first                 '?'
_journal_page_last                  '?'
_journal_year                       '2002'
_ccdc_journal_depnumber             '?'



#==============================================================================

# 4. TEXT


_publ_section_references
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Altomare,A., Cascarano,G., Giacovazzo,C., Guagliardi,A.,
```

Moliterni,.A,G,G., Burla,M.C., Polidori,G., Camalli,M.
& Spagna,R. (1997). SIR97. University of Bari, Italy.

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Oak Ridge National Laboratory, Tennessee, USA.

Busing,W.R., Martin,K.O. & Levy,H.A. (1962). ORFLS. Report
ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.

Hazell,A. (1995). KRYSTAL, An integrated system of
crystallographic programs. Aarhus University, Denmark.

Rogers,D. (1981). Acta Cryst. A37, 734-741.

Siemens (1995) SMART, SAINT and XPREP Area-Detector Control and
Integration Software. Siemens Analytical X-ray Instruments Inc.
Madison, Wisconsin, USA.

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# 5. CHEMICAL DATA

_chemical_name_systematic
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4-(4-Chloro-phenyl)-5-methyl-5-nitro-hexan-2-one
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_chemical_formula_sum                C13H16ClNO3
_chemical_formula_weight             269.74

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# 6. CRYSTAL DATA

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_cell_length_b                       10.869(4)
_cell_length_c                       20.746(8)
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_cell_angle_beta                     90.000
_cell_angle_gamma                    90.000
_cell_volume                         1347(1)
_cell_formula_units_Z                4
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_diffrn_measurement_method           '\w rotation scans with narrow frames'
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      ORTEP-III (Burnett & Johnson,  1996), KRYSTAL
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_atom_type_scat_source               IntTabIV

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_refine_ls_extinction_method
;
 Type 1 Lorentzian isotropic (Becker and Coppens, 1974)
;
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; Least squares refinement included a factor according to Rogers
  (1982). The 2107 significant reflections used included 826
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_refine_ls_abs_structure_Rogers     0.83(15)
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_refine_ls_number_parameters        229
_refine_ls_number_restraints        0
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_refine_ls_wR_factor_ref            0.041
_refine_ls_goodness_of_fit_ref      1.25
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_refine_ls_shift/su_mean            0.0001
_refine_diff_density_max            0.47(8)
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# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

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_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_thermal_displace_type
  CL1     0.0406(1)   -0.08628(6)   0.45928(4)   0.0336(4)     Uij
  O2     -0.4569(3)   -0.4202(2)    0.22104(9)   0.033(1)      Uij
  C3     -0.2822(4)   -0.5519(2)    0.3301(1)    0.019(1)      Uij
  O4     -0.1166(4)   -0.3639(2)    0.2078(1)    0.044(1)      Uij
  C5     -0.0596(5)   -0.2239(2)    0.4260(1)    0.023(1)      Uij
  N6     -0.2556(4)   -0.4361(2)    0.2275(1)    0.022(1)      Uij
  C7     -0.1787(4)   -0.5560(2)    0.2607(1)    0.021(1)      Uij
  C8     -0.3392(4)   -0.3358(2)    0.3689(1)    0.020(1)      Uij
  C9     -0.2686(5)   -0.2270(2)    0.3981(1)    0.023(1)      Uij
  C10    -0.4365(5)   -0.7115(2)    0.4085(1)    0.026(1)      Uij
  C11    -0.2798(5)   -0.6572(3)    0.2193(2)    0.027(2)      Uij
  O12    -0.5984(4)   -0.6467(2)    0.4160(1)    0.052(1)      Uij
  C13    -0.2047(4)   -0.4401(2)    0.3678(1)    0.020(1)      Uij
  C14     0.0755(5)   -0.3274(3)    0.4284(1)    0.026(2)      Uij
  C15     0.0027(4)   -0.4349(2)    0.3994(1)    0.024(1)      Uij
  C16    -0.2463(5)   -0.6752(2)    0.3648(2)    0.026(2)      Uij
  C17     0.0759(5)   -0.5597(3)    0.2590(2)    0.027(2)      Uij
  C18    -0.4160(6)   -0.8340(3)    0.4416(2)    0.040(2)      Uij
  H3     -0.434(4)   -0.546(2)    0.324(1)    0.010(6)      Uiso
  H8     -0.487(4)   -0.340(2)    0.348(1)   -0.001(5)      Uiso
  H9     -0.357(4)   -0.151(2)    0.397(1)    0.025(7)      Uiso
  H11a   -0.244(5)   -0.646(2)    0.171(2)    0.031(8)      Uiso
  H11b   -0.208(5)   -0.725(3)    0.230(2)    0.045(10)     Uiso
  H11c   -0.439(6)   -0.664(3)    0.226(1)    0.039(8)      Uiso
  H14     0.211(4)   -0.322(2)    0.447(1)    0.015(7)      Uiso
  H15     0.097(5)   -0.505(2)    0.402(1)    0.014(6)      Uiso
  H16a   -0.211(6)   -0.742(3)    0.334(2)    0.052(10)     Uiso
  H16b   -0.124(5)   -0.678(2)    0.391(1)    0.027(8)      Uiso
  H17a    0.126(4)   -0.559(2)    0.213(1)    0.027(8)      Uiso
  H17b    0.140(5)   -0.488(3)    0.282(1)    0.034(8)      Uiso
  H17c    0.123(6)   -0.635(3)    0.285(1)    0.053(10)     Uiso
  H18a   -0.570(8)   -0.868(4)    0.459(2)    0.084(12)     Uiso
  H18b   -0.367(7)   -0.900(4)    0.411(2)    0.081(13)     Uiso
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  H18c     -0.339(7)   -0.821(4)    0.477(2)    0.077(15)     Uiso

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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
CL1    0.0360(4)   0.0278(3)   0.0371(4)  -0.0069(3)  -0.0014(3)  -0.0080(3)
O2     0.024(1)    0.034(1)    0.042(1)    0.005(1)   -0.001(1)    0.011(1)
C3     0.008(1)    0.020(1)    0.030(2)    0.003(1)    0.000(1)    0.005(1)
O4     0.036(1)    0.035(1)    0.061(2)   -0.015(1)    0.001(1)    0.017(1)
C5     0.025(1)    0.021(1)    0.022(1)   -0.003(1)    0.000(1)   -0.004(1)
N6     0.024(1)    0.018(1)    0.025(1)   -0.004(1)    0.002(1)   -0.003(1)
C7     0.015(1)    0.016(1)    0.032(2)    0.000(1)    0.001(1)   -0.002(1)
C8     0.019(1)    0.020(1)    0.020(2)    0.004(1)   -0.002(1)    0.001(1)
C9     0.025(1)    0.018(1)    0.026(2)    0.006(1)    0.003(1)    0.001(1)
C10    0.028(1)    0.020(1)    0.029(2)    0.005(1)    0.002(1)    0.004(1)
C11    0.018(2)    0.025(2)    0.040(2)   -0.002(1)    0.000(1)   -0.008(1)
O12    0.039(1)    0.046(1)    0.071(2)    0.024(1)    0.032(1)    0.029(1)
C13    0.017(1)    0.021(1)    0.021(1)    0.003(1)    0.004(1)    0.002(1)
C14    0.014(1)    0.037(2)    0.027(2)    0.002(1)   -0.002(1)   -0.003(1)
C15    0.018(1)    0.025(1)    0.028(2)    0.007(1)    0.002(1)    0.000(1)
C16    0.017(1)    0.021(1)    0.039(2)    0.007(1)   -0.001(1)    0.006(1)
C17    0.019(1)    0.029(2)    0.034(2)   -0.001(1)    0.005(1)   -0.006(1)
C18    0.043(2)    0.029(2)    0.047(2)    0.002(2)    0.001(2)    0.016(2)

#================================

# 10. MOLECULAR GEOMETRY

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 CL1      C5        1.753(3)
 O2       N6        1.222(3)
 C3       C13       1.517(4)
 C3       C16       1.537(4)
 C3       C7        1.567(4)
 O4       N6        1.213(3)
 C5       C9        1.376(4)
 C5       C14       1.385(4)
 N6       C7        1.544(4)
 C7       C11       1.521(4)
 C7       C17       1.522(4)
 C8       C13       1.390(4)
 C8       C9        1.393(4)
 C10      O12       1.207(3)
 C10      C18       1.503(4)
 C10      C16       1.507(4)
 C13      C15       1.403(4)
 C14      C15       1.385(4)
 C3       H3        0.921(25)
 C8       H8        0.984(21)
 C9       H9        0.984(26)
 C11      H11b      0.886(33)
 C11      H11c      0.966(34)
 C11      H11a      1.031(31)
 C14      H14       0.899(26)
 C15      H15       0.947(27)
 C16      H16b      0.914(29)
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C16     H16a     0.984(34)
C17     H17b     0.991(30)
C17     H17a     0.994(28)
C17     H17c     1.023(35)
C18     H18c     0.873(42)
C18     H18b     1.000(44)
C18     H18a     1.058(49)

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   _geom_angle_atom_site_label_2
   _geom_angle_atom_site_label_3
   _geom_angle
C13     C3      C16       114.5(2)
C7      C3      C13       112.0(2)
C7      C3      C16       110.5(2)
C9      C5      C14       121.6(3)
CL1     C5      C9        119.7(2)
CL1     C5      C14       118.6(2)
O2      N6      O4        123.1(2)
O4      N6      C7        119.5(2)
O2      N6      C7        117.4(2)
C11     C7      C17       111.3(2)
N6      C7      C11       103.9(2)
C3      C7      C11       112.5(2)
N6      C7      C17       108.0(2)
C3      C7      C17       114.7(2)
C3      C7      N6        105.6(2)
C9      C8      C13       121.6(2)
C5      C9      C8        118.6(3)
O12     C10     C18       121.6(3)
O12     C10     C16       121.9(2)
C16     C10     C18       116.4(3)
C8      C13     C15       118.0(2)
C3      C13     C8        119.1(2)
C3      C13     C15       122.8(2)
C5      C14     C15       119.1(3)
C13     C15     C14       121.0(2)
C3      C16     C10       113.9(2)
C13     C3      H3        108.9(15)
C16     C3      H3        105.1(15)
C7      C3      H3        105.1(15)
C13     C8      H8        118.6(12)
C9      C8      H8        119.8(12)
C5      C9      H9        118.5(16)
C8      C9      H9        122.8(16)
H11b    C11     H11c      112.1(28)
H11a    C11     H11b      103.9(26)
C7      C11     H11b      105.8(22)
H11a    C11     H11c      111.3(24)
C7      C11     H11c      111.1(18)
C7      C11     H11a      112.3(15)
C15     C14     H14       121.5(16)
C5      C14     H14       119.4(16)
C14     C15     H15       117.9(15)
C13     C15     H15       121.1(15)
H16a    C16     H16b      100.9(26)
C10     C16     H16b      103.8(18)
C3      C16     H16b      114.5(17)
C10     C16     H16a      110.7(20)
C3      C16     H16a      112.0(19)
H17a    C17     H17b      110.1(23)
H17b    C17     H17c      104.9(24)
C7      C17     H17b      110.8(17)
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  H17a    C17     H17c       115.6(25)
  C7      C17     H17a       108.9(16)
  C7      C17     H17c       106.4(20)
  H18b    C18     H18c         120(4)
  H18a    C18     H18c       102.7(32)
  C10     C18     H18c       106.3(30)
  H18a    C18     H18b       103.5(30)
  C10     C18     H18b       111.3(23)
  C10     C18     H18a       113.2(22)

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data_cpd-5b

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# 5. CHEMICAL DATA

_chemical_name_systematic
;
cis-5-Methyl-2,3-diphenyl-pyrrolidine
;
_chemical_formula_sum                C17H19N
_chemical_formula_weight             237.35

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# 6. CRYSTAL DATA

_symmetry_cell_setting               monoclinic
_symmetry_space_group_name_H-M       'P21/n'

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   '-x,-y,-z'
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_cell_length_a                       13.7301(8)
_cell_length_b                       6.1319(4)
_cell_length_c                       16.064(1)
_cell_angle_alpha                    90.000
_cell_angle_beta                     102.584(1)
_cell_angle_gamma                    90.000
_cell_volume                         1320.0(1)
_cell_formula_units_Z                4
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_cell_measurement_reflns_used        4884
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# 7. EXPERIMENTAL DATA

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_diffrn_radiation_monochromator      'graphite'
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_diffrn_measurement_method           '\w rotation scans with narrow frames'
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_diffrn_reflns_reduction_process     12427
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      ORTEP-III (Burnett & Johnson,  1996), KRYSTAL
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_computing_publication_material       KRYSTAL
_atom_type_scat_source               IntTabIV

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# 8. REFINEMENT DATA

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_refine_ls_hydrogen_treatment        refall
_refine_ls_extinction_method
;
 Type 1 Lorentzian isotropic (Becker and Coppens, 1974)
;
_refine_ls_extinction_coef           34(13)
_refine_ls_number_reflns             2695
_refine_ls_number_parameters         240
_refine_ls_number_restraints         0
_refine_ls_number_constraints        0
_refine_ls_R_factor_gt               0.038
_refine_ls_wR_factor_ref             0.047
_refine_ls_goodness_of_fit_ref       1.48
_refine_ls_shift/su_max              0.0003
_refine_ls_shift/su_mean             0.00003
```

```
_refine_diff_density_max         0.34(4)
_refine_diff_density_min         -0.22(4)
#==================================

data_cpd-5b

#==================================

# 5. CHEMICAL DATA

_chemical_name_systematic
;
cis-5-Methyl-2,3-diphenyl-pyrrolidine
;
_chemical_formula_sum            C17H19N
_chemical_formula_weight         237.35

#==================================
# 6. CRYSTAL DATA

_symmetry_cell_setting           monoclinic
_symmetry_space_group_name_H-M   'P21/n'

loop_
_symmetry_equiv_pos_as_xyz
   '+x,+y,+z'
   '1/2-x,1/2+y,1/2-z'
   '-x,-y,-z'
   '1/2+x,1/2-y,1/2+z'

_cell_length_a                   13.7301(8)
_cell_length_b                   6.1319(4)
_cell_length_c                   16.064(1)
_cell_angle_alpha                90.000
_cell_angle_beta                 102.584(1)
_cell_angle_gamma                90.000
_cell_volume                     1320.0(1)
_cell_formula_units_Z            4
_cell_measurement_temperature    120
_cell_measurement_reflns_used    4884
_cell_measurement_theta_min      2.6
_cell_measurement_theta_max      29.8
_exptl_crystal_description       'thick plate'
_exptl_crystal_colour            colourless
_exptl_crystal_size_max          0.54
_exptl_crystal_size_mid          0.40
_exptl_crystal_size_min          0.16
_exptl_crystal_density_meas      'not measured'
_exptl_crystal_density_diffrn    1.194
_exptl_crystal_density_method    'not measured'
_exptl_crystal_F_000              512
_exptl_absorpt_coefficient_mu    0.069
_exptl_absorpt_correction_type   'none'

#==================================

# 7. EXPERIMENTAL DATA

_diffrn_ambient_temperature      120
_diffrn_radiation_wavelength     0.71073
_diffrn_radiation_type           'Mo K\a'
_diffrn_radiation_source         'x-ray tube'
_diffrn_radiation_monochromator  'graphite'
_diffrn_measurement_device       'Siemens SMART CCD diffractometer'
```

```
_diffrn_measurement_method           '\w rotation scans with narrow frames'
_diffrn_reflns_number                12427
_diffrn_reflns_av_R_equivalents      0.052
_diffrn_reflns_limit_h_min           -19
_diffrn_reflns_limit_h_max           19
_diffrn_reflns_limit_k_min           -8
_diffrn_reflns_limit_k_max           8
_diffrn_reflns_limit_l_min           -22
_diffrn_reflns_limit_l_max           19
_diffrn_reflns_theta_min             2.6
_diffrn_reflns_theta_max             29.8
_diffrn_reflns_reduction_process     12427
_reflns_number_total                 4078
_reflns_number_gt                    2695
_reflns_threshold_expression         I>3\s(I)
_computing_data_collection           'SMART (Siemens, 1995)'
_computing_cell_refinement           'SAINT (Siemens, 1995)'
_computing_data_reduction            'SAINT (Siemens, 1995)'
_computing_structure_solution
;
     SIR97 (Altomare et al., 1997 ), KRYSTAL
;
_computing_structure_refinement      'modified ORFLS(1962), KRYSTAL'
_computing_molecular_graphics
;
      ORTEP-III (Burnett & Johnson,  1996), KRYSTAL
;
_computing_publication_material       KRYSTAL
_atom_type_scat_source               IntTabIV

#===================================

# 8. REFINEMENT DATA

_refine_ls_structure_factor_coef     F
_refine_ls_matrix_type               full
_refine_ls_weighting_scheme          calc
_refine_ls_weighting_details
;
w= 1/{[\s~cs~(F^2^)+1.03F^2^]^1/2^- |F|}^2^
;
_refine_ls_hydrogen_treatment        refall
_refine_ls_extinction_method
;
 Type 1 Lorentzian isotropic (Becker and Coppens, 1974)
;
_refine_ls_extinction_coef           34(13)
_refine_ls_number_reflns             2695
_refine_ls_number_parameters         240
_refine_ls_number_restraints         0
_refine_ls_number_constraints        0
_refine_ls_R_factor_gt               0.038
_refine_ls_wR_factor_ref             0.047
_refine_ls_goodness_of_fit_ref       1.48
_refine_ls_shift/su_max              0.0003
_refine_ls_shift/su_mean             0.00003
_refine_diff_density_max             0.34(4)
_refine_diff_density_min             -0.22(4)

 #=========================================

 # 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

 loop_
```

```
_atom_site_label
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_thermal_displace_type
  N1       0.61769(7)  -0.0354(2)   0.41675(6)   0.0212(5)     Uij
  C1       0.78113(10) -0.2081(2)   0.41733(9)   0.0300(7)     Uij
  C2       0.72525(8)   0.0019(2)   0.42377(7)   0.0221(6)     Uij
  C3       0.72646(8)   0.1663(2)   0.35199(8)   0.0242(6)     Uij
  C4       0.62267(8)   0.2758(2)   0.33229(7)   0.0202(6)     Uij
  C5       0.57518(8)   0.1845(2)   0.40502(7)   0.0200(6)     Uij
  C6       0.46283(8)   0.1957(2)   0.38791(7)   0.0198(6)     Uij
  C7       0.40192(9)   0.0193(2)   0.35744(7)   0.0229(6)     Uij
  C8       0.29847(9)   0.0402(2)   0.34062(8)   0.0260(6)     Uij
  C9       0.25527(9)   0.2379(2)   0.35311(8)   0.0272(6)     Uij
  C10      0.31516(9)   0.4151(2)   0.38325(8)   0.0269(7)     Uij
  C11      0.41819(9)   0.3938(2)   0.40037(7)   0.0236(6)     Uij
  C12      0.56049(7)   0.2271(2)   0.24422(7)   0.0189(5)     Uij
  C13      0.55690(9)   0.0200(2)   0.20775(7)   0.0225(6)     Uij
  C14      0.49809(9)  -0.0199(2)   0.12720(8)   0.0252(6)     Uij
  C15      0.44131(9)   0.1454(2)   0.08197(8)   0.0260(6)     Uij
  C16      0.44368(9)   0.3513(2)   0.11757(8)   0.0267(6)     Uij
  C17      0.50273(8)   0.3912(2)   0.19810(8)   0.0234(6)     Uij
  H1n      0.6062(10) -0.098(2)    0.4650(9)    0.031(4)      Uiso
  H1a      0.8506(11) -0.176(2)    0.4226(9)    0.040(4)      Uiso
  H1b      0.7555(10) -0.280(2)    0.3606(10)   0.035(4)      Uiso
  H1c      0.7740(11) -0.313(2)    0.4631(10)   0.046(4)      Uiso
  H2       0.7538(9)   0.075(2)    0.4797(8)    0.025(3)      Uiso
  H3a      0.7407(10)  0.092(2)    0.3016(9)    0.039(4)      Uiso
  H3b      0.7811(10)  0.277(2)    0.3688(9)    0.036(4)      Uiso
  H4       0.6296(9)   0.436(2)    0.3388(8)    0.025(3)      Uiso
  H5       0.6023(8)   0.275(2)    0.4564(8)    0.017(3)      Uiso
  H7       0.4327(9)  -0.123(2)    0.3497(8)    0.023(3)      Uiso
  H8       0.2562(10) -0.081(2)    0.3190(8)    0.029(3)      Uiso
  H9       0.1841(10)  0.257(2)    0.3398(9)    0.034(4)      Uiso
  H10      0.2848(10)  0.556(2)    0.3916(9)    0.037(4)      Uiso
  H11      0.4598(9)   0.520(2)    0.4202(8)    0.023(3)      Uiso
  H13      0.5963(9)  -0.099(2)    0.2381(8)    0.025(3)      Uiso
  H14      0.4970(10) -0.165(2)    0.1023(9)    0.034(4)      Uiso
  H15      0.3998(9)   0.119(2)    0.0255(8)    0.028(3)      Uiso
  H16      0.4040(10)  0.468(2)    0.0864(9)    0.032(4)      Uiso
  H17      0.5042(10)  0.540(2)    0.2230(8)    0.030(4)      Uiso

loop_
_atom_site_aniso_label
_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
 N1   0.0203(5)  0.0238(5)  0.0203(5)   0.0024(4)   0.0063(4)   0.0045(4)
 C1   0.0252(6)  0.0340(7)  0.0322(7)   0.0069(5)   0.0089(5)   0.0033(6)
 C2   0.0185(5)  0.0278(6)  0.0191(5)   0.0009(4)   0.0023(4)   0.0000(5)
 C3   0.0186(5)  0.0284(6)  0.0256(6)  -0.0024(5)   0.0044(4)   0.0029(5)
 C4   0.0201(5)  0.0186(5)  0.0211(6)  -0.0028(4)   0.0024(4)   0.0002(4)
 C5   0.0204(5)  0.0215(6)  0.0176(5)   0.0004(4)   0.0032(4)  -0.0022(4)
 C6   0.0213(5)  0.0238(6)  0.0150(5)   0.0010(4)   0.0056(4)   0.0006(4)
 C7   0.0242(6)  0.0227(6)  0.0231(6)   0.0010(5)   0.0076(4)  -0.0017(5)
 C8   0.0241(6)  0.0298(6)  0.0243(6)  -0.0042(5)   0.0057(5)   0.0003(5)
 C9   0.0205(6)  0.0358(7)  0.0258(6)   0.0037(5)   0.0065(5)   0.0057(5)
 C10  0.0283(6)  0.0265(6)  0.0274(6)   0.0081(5)   0.0090(5)   0.0033(5)
 C11  0.0267(6)  0.0224(6)  0.0217(6)   0.0003(5)   0.0054(4)  -0.0007(5)
```

```
 C12   0.0168(5)   0.0217(5)   0.0194(5)   -0.0034(4)    0.0064(4)    0.0013(4)
 C13   0.0248(6)   0.0209(6)   0.0227(6)   -0.0004(4)    0.0071(4)    0.0019(5)
 C14   0.0283(6)   0.0245(6)   0.0245(6)   -0.0058(5)    0.0094(5)   -0.0039(5)
 C15   0.0215(5)   0.0352(7)   0.0205(6)   -0.0053(5)    0.0030(4)   -0.0013(5)
 C16   0.0219(6)   0.0314(7)   0.0252(6)    0.0028(5)    0.0014(5)    0.0034(5)
 C17   0.0229(5)   0.0226(6)   0.0245(6)    0.0014(4)    0.0047(4)   -0.0005(5)

#================================

# 10. MOLECULAR GEOMETRY

loop_
    _geom_bond_atom_site_label_1
    _geom_bond_atom_site_label_2
    _geom_bond_distance
 N1       C5         1.465(2)
 N1       C2         1.474(2)
 C1       C2         1.514(2)
 C2       C3         1.534(2)
 C3       C4         1.545(2)
 C4       C12        1.514(2)
 C4       C5         1.561(2)
 C5       C6         1.508(2)
 C6       C7         1.390(2)
 C6       C11        1.395(2)
 C7       C8         1.393(2)
 C8       C9         1.383(2)
 C9       C10        1.385(2)
 C10      C11        1.387(2)
 C12      C17        1.391(2)
 C12      C13        1.395(2)
 C13      C14        1.390(2)
 C14      C15        1.385(2)
 C15      C16        1.383(2)
 C16      C17        1.390(2)
 N1       H1n        0.910(15)
 C1       H1a        0.959(15)
 C1       H1c        0.998(17)
 C1       H1b        1.005(15)
 C2       H2         1.003(13)
 C3       H3a        0.985(16)
 C3       H3b        1.003(14)
 C4       H4         0.990(14)
 C5       H5         0.997(12)
 C7       H7         0.990(14)
 C8       H8         0.961(14)
 C9       H9         0.960(14)
 C10      H10        0.983(15)
 C11      H11        0.973(13)
 C13      H13        0.973(13)
 C14      H14        0.972(15)
 C15      H15        0.974(13)
 C16      H16        0.971(14)
 C17      H17        0.997(14)

loop_
    _geom_angle_atom_site_label_1
    _geom_angle_atom_site_label_2
    _geom_angle_atom_site_label_3
    _geom_angle
 C2       N1       C5         103.39(9)
 N1       C2       C1          112.2(1)
 N1       C2       C3         102.65(9)
 C1       C2       C3          114.7(1)
```

C2	C3	C4	106.2(1)
C3	C4	C12	114.4(1)
C5	C4	C12	112.73(9)
C3	C4	C5	102.01(9)
N1	C5	C6	115.4(1)
N1	C5	C4	101.92(9)
C4	C5	C6	115.13(9)
C7	C6	C11	118.6(1)
C5	C6	C7	122.8(1)
C5	C6	C11	118.5(1)
C6	C7	C8	120.4(1)
C7	C8	C9	120.3(1)
C8	C9	C10	119.8(1)
C9	C10	C11	119.9(1)
C6	C11	C10	121.0(1)
C13	C12	C17	118.1(1)
C4	C12	C17	119.7(1)
C4	C12	C13	122.2(1)
C12	C13	C14	120.8(1)
C13	C14	C15	120.4(1)
C14	C15	C16	119.4(1)
C15	C16	C17	120.1(1)
C12	C17	C16	121.2(1)
C5	N1	H1n	111.1(9)
C2	N1	H1n	110.9(8)
H1a	C1	H1c	108.9(12)
H1a	C1	H1b	108.4(12)
C2	C1	H1a	109.0(9)
H1b	C1	H1c	108.1(12)
C2	C1	H1c	111.5(9)
C2	C1	H1b	110.8(8)
N1	C2	H2	109.1(7)
C1	C2	H2	109.6(7)
C3	C2	H2	108.3(8)
H3a	C3	H3b	105.2(12)
C2	C3	H3a	110.6(9)
C4	C3	H3a	111.8(8)
C2	C3	H3b	111.7(8)
C4	C3	H3b	111.3(8)
C12	C4	H4	108.4(7)
C3	C4	H4	110.3(8)
C5	C4	H4	108.9(8)
N1	C5	H5	109.8(7)
C6	C5	H5	107.9(7)
C4	C5	H5	106.2(7)
C6	C7	H7	119.4(7)
C8	C7	H7	120.2(7)
C9	C8	H8	119.2(8)
C7	C8	H8	120.5(8)
C8	C9	H9	121.4(9)
C10	C9	H9	118.8(9)
C9	C10	H10	120.1(8)
C11	C10	H10	120.0(9)
C10	C11	H11	119.4(8)
C6	C11	H11	119.6(7)
C14	C13	H13	118.8(8)
C12	C13	H13	120.4(8)
C15	C14	H14	119.8(8)
C13	C14	H14	119.8(8)
C16	C15	H15	119.6(8)
C14	C15	H15	121.0(8)
C15	C16	H16	120.1(8)
C17	C16	H16	119.8(8)
C16	C17	H17	119.4(8)

C12	C17	H17	119.4(8)

X-Ray Structure of *cis*-5-Methyl-2,3-Diphenyl-Pyrrolidine, 5b.