University of Groningen

Cycloruthenated Primary and Secondary Amines as Efficient Catalyst Precursors for Asymmetric Transfer Hydrogenation

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_audit_creation_date                    6-10-03

# 1. SUBMISSION DETAILS

_publ_contact_author_name               # Name of author for correspondance
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_publ_requested_journal                 'Angew. Chem. Int. Ed.'
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;
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_publ_requested_category                FM
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# 2. PROCESSING SUMMARY (IUCr Office Use Only)

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_journal_date_to_coeditor          ?
_journal_date_from_coeditor        ?
_journal_date_accepted             ?

_journal_date_printers_first       ?
_journal_date_printers_final       ?
_journal_date_proofs_out           ?
_journal_date_proofs_in            ?


_journal_coeditor_name             ?
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_journal_techeditor_code           ?
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_journal_coden_ASTM                ?
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_journal_issue                     ?
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_journal_page_last                 ?

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# 3. TITLE AND AUTHOR LIST

_publ_section_title
; Cycloruthenated primary and secondary amines as efficient catalyst
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;


# The loop structure below should contain the names  and addresses of all
# authors, in the required order  of publication. Repeat as necessary.

loop_
_publ_author_name
_publ_author_address
'Dupont Karl'            #<--'Last name, first name'
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 Universite Louis Pasteur
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 67000 Strasbourg
 France
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4. TEXT

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_publ_section_exptl_refinement
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_publ_section_references
;

Burla, M.C., Camalli, M., Cascarano, G.,Giacovazzo, C., Polidori, G.,
Spagna, R. &Viterbo, D. (1989). SIR. J. Appl. Cryst. 22, 389-393.

OpenMoleN, Interactive Intelligent Structure solution (1997)
Nonius B.V., Delft, The Netherlands.

KappaCCD Operation Manual (1997), Nonius B.V., Delft, The Nederlands.

Otwinowski, Z. & Minor, W. (1997), Methods in Enzymology, 276, 307-326.

Walker, N. & Stuart, D. (1983). Acta Cryst., A39, 158-166.

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 # If more than one structure is reported, sections 5-10 should be filled in
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data_st1440

#===

5. CHEMICAL DATA

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_chemical_name_common   ?
_chemical_formula_moiety   'C16 H19 F6 N2 P Ru'
_chemical_formula_structural   ?
_chemical_formula_analytical   ?
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_chemical_formula_weight   485.38
_chemical_melting_point   ?
_chemical_compound_source   ?

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     _atom_type_scat_dispersion_imag
     _atom_type_scat_source
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P  ?  0.090  0.095 International_Tables_Vol_IV_Table_2.3.1
Ru ? -1.420  0.836 International_Tables_Vol_IV_Table_2.3.1


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_symmetry_space_group_name_Hall     ' P 2ac 2ab'
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    _symmetry_equiv_pos_as_xyz
    'x,y,z'
    1/2-x,-y,1/2+z
    -x,1/2+y,1/2-z
    1/2+x,1/2-y,-z
_cell_length_a                      9.2385(2)
_cell_length_b                      12.0517(3)
_cell_length_c                      16.4150(5)
_cell_angle_alpha                   90
_cell_angle_beta                    90
_cell_angle_gamma                   90
_cell_volume                        1827.64(8)
_cell_formula_units_Z               4
_cell_measurement_temperature       173
_cell_measurement_reflns_used       5241
_cell_measurement_theta_min          1.4
_cell_measurement_theta_max         30.0
_cell_special_details
;
Cell parameters refined using Scalepack part of DENZO
 Z. Otwinowski, W. Minor, 1997
;

_exptl_crystal_description          prism
_exptl_crystal_colour               'yellow'
_exptl_crystal_size_max             0.20
_exptl_crystal_size_mid             0.20
_exptl_crystal_size_min             0.10
_exptl_crystal_density_meas         ?
_exptl_crystal_density_diffrn       1.76
_exptl_crystal_density_method       'none'

_exptl_crystal_F_000                968
_exptl_absorpt_coefficient_mu       1.005
_exptl_absorpt_correction_type      refdelf_(Walker_&_Stuart,_1983)
_exptl_absorpt_correction_T_min     0.818
_exptl_absorpt_correction_T_max     0.904


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_diffrn_special_details
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_diffrn_ambient_temperature         173
_diffrn_radiation_wavelength        0.71073
_diffrn_radiation_type              Mo-K\a

_diffrn_source                      xray_tube
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_diffrn_radiation_monochromator      graphite
_diffrn_measurement_device_type      KappaCCD
_diffrn_measurement_method              '\p scans'

_diffrn_measurement_details
;
   5241 reflections were collected using program Collect
 ("Collect" Data collection software, Nonius B.V., 1998)
 The conditions were as follow : crystal to detector distance =  36. mm.
 Scan angle =  2.0 deg 1 scans of   30 sec per frame.
 Data collection was divided into  2 set(s)
 with the following starting angles and number of frames :

 Set 1 Theta =    8.80 Omega =    0.00 Kappa =    0.00   73 frames
 Set 2 Theta =   -6.60 Kappa = 121.00 Phi  =    0.00   14 frames
 Friedel pairs were averaged. Internal R = 0.04
;

_diffrn_standards_number             ?
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_diffrn_standards_interval_time      ?
_diffrn_standards_decay_%            0

_diffrn_reflns_number                5241
_diffrn_reflns_av_R_equivalents      0.040
_diffrn_reflns_av_sigmaI/netI        0.333
_diffrn_reflns_limit_h_min           -12
_diffrn_reflns_limit_h_max           13
_diffrn_reflns_limit_k_min           -16
_diffrn_reflns_limit_k_max           16
_diffrn_reflns_limit_l_min           -22
_diffrn_reflns_limit_l_max           23
_diffrn_reflns_theta_min             2.5
_diffrn_reflns_theta_max             30.04

_reflns_number_total                 5241
_reflns_number_gt                    3497
_reflns_threshold_expression         >3.0\s(I)

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_computing_structure_refinement      LSFM_OpenMoleN_(_1997)
_computing_molecular_graphics        ?
_computing_publication_material      CIFGEN_IN_OpenMoleN_(_1997)

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_refine_ls_extinction_coef          ?
_refine_ls_abs_structure_Flack      -0.01(5)
_refine_ls_number_reflns            3497
_refine_ls_number_parameters        235
_refine_ls_number_restraints        0
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_refine_ls_abs_structure_details    'Flack H D  (1983), Acta Cryst. A39, 876-881'
_refine_ls_R_factor_all             0.071
_refine_ls_R_factor_gt              0.036
_refine_ls_wR_factor_all            0.123
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_refine_ls_wR_factor_ref          0.044
_refine_ls_goodness_of_fit_all     3.376
_refine_ls_goodness_of_fit_ref     1.066
_refine_ls_shift/su_max           0.009
_refine_ls_shift/esd_mean         0.002
_refine_diff_density_max          0.789
_refine_diff_density_min          -0.472


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

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

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_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_adp_type
_atom_site_calc_flag
_atom_site_calc_attached_atom
_atom_site_type_symbol
RU   0.29861(3)   0.13386(3)   0.71884(2)   0.0249(1)   Uani  ? ?  Ru
N1   0.2897(4)    0.0057(3)    0.6354(2)    0.026(2)    Uani  ? ?  N
C1   0.2984(5)    -0.0576(3)   0.5841(3)    0.028(2)    Uani  ? ?  C
C2   0.3112(5)    -0.1381(4)   0.5190(3)    0.042(2)    Uani  ? ?  C
N2   0.0745(3)    0.1536(3)    0.6950(2)    0.027(2)    Uani  ? ?  N
C3   -0.0211(4)   0.1232(4)    0.7673(3)    0.032(2)    Uani  ? ?  C
C4   -0.0517(5)   0.2250(5)    0.8186(3)    0.043(3)    Uani  ? ?  C
C5   0.0556(4)    0.0333(4)    0.8124(3)    0.027(2)    Uani  ? ?  C
C6   0.2080(4)    0.0238(3)    0.8006(2)    0.021(2)    Uani  ? ?  C
C7   0.2782(4)    -0.0570(4)   0.8442(3)    0.032(2)    Uani  ? ?  C
C8   0.2068(5)    -0.1258(4)   0.8978(3)    0.038(2)    Uani  ? ?  C
C9   0.0612(5)    -0.1146(4)   0.9101(3)    0.036(2)    Uani  ? ?  C
C10  -0.0156(4)   -0.0345(4)   0.8664(3)    0.032(2)    Uani  ? ?  C
C11  0.3397(6)    0.3115(4)    0.7242(4)    0.046(3)    Uani  ? ?  C
C12  0.3619(5)    0.2692(5)    0.8009(3)    0.046(3)    Uani  ? ?  C
C13  0.4570(5)    0.1823(5)    0.8094(3)    0.050(3)    Uani  ? ?  C
C14  0.5293(5)    0.1399(5)    0.7465(5)    0.055(3)    Uani  ? ?  C
C15  0.5076(6)    0.1818(6)    0.6679(4)    0.081(3)    Uani  ? ?  C
C16  0.4127(7)    0.2728(5)    0.6564(4)    0.069(3)    Uani  ? ?  C
P    -0.3490(1)   0.0167(1)    1.02425(8)   0.0285(6)   Uani  ? ?  P
F1   -0.4544(3)   0.1218(3)    1.0249(2)    0.063(2)    Uani  ? ?  F
F2   -0.2460(3)   -0.0885(3)   1.0221(2)    0.051(2)    Uani  ? ?  F
F3   -0.3548(2)   0.0174(2)    0.9274(2)    0.037(1)    Uani  ? ?  F
F4   -0.3441(3)   0.0176(3)    1.1213(2)    0.053(2)    Uani  ? ?  F
F5   -0.4867(3)   -0.0629(3)   1.0270(2)    0.047(2)    Uani  ? ?  F
F6   -0.2124(3)   0.0975(3)    1.0216(2)    0.054(2)    Uani  ? ?  F
H1   0.2174   -0.1584   0.5006   0.0565   Uiso   calc   C2    H
H2   0.3643   -0.1067   0.4752   0.0565   Uiso   calc   C2    H
H3   0.3601   -0.2021   0.5386   0.0565   Uiso   calc   C2    H
H4   -0.1136   0.0969   0.7510   0.0428   Uiso   calc   C3    H
H5   -0.1113   0.2050   0.8634   0.0569   Uiso   calc   C4    H
H6   0.0370   0.2547   0.8382   0.0569   Uiso   calc   C4    H
H7   -0.0998   0.2790   0.7863   0.0569   Uiso   calc   C4    H
H8   0.3796   -0.0659   0.8371   0.0415   Uiso   calc   C7    H
H9   0.2590   -0.1814   0.9265   0.0501   Uiso   calc   C8    H
H10  0.0126   -0.1609   0.9481   0.0477   Uiso   calc   C9    H
H11  -0.1170   -0.0270   0.8739   0.0414   Uiso   calc   C10   H
H12  0.2714   0.3697   0.7175   0.0804   Uiso   calc   C11   H
H13  0.3128   0.2991   0.8467   0.0609   Uiso   calc   C12   H
H14  0.4718   0.1514   0.8620   0.0658   Uiso   calc   C13   H
H15  0.5959   0.0809   0.7550   0.0900   Uiso   calc   C14   H
H16  0.5561   0.1495   0.6227   0.1072   Uiso   calc   C15   H
H17  0.3999   0.3059   0.6043   0.0962   Uiso   calc   C16   H
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N1  0.026(2)  0.026(2)  0.027(2)  -0.000(2)  0.006(2)  0.001(2)  N
C1  0.029(2)  0.024(2)  0.031(2)  -0.002(2)  0.009(2)  0.002(2)  C
C2  0.060(3)  0.034(2)  0.036(2)  0.002(3)  0.013(2)  -0.009(3)  C
N2  0.023(2)  0.026(2)  0.031(2)  0.003(2)  0.003(1)  -0.005(2)  N
C3  0.022(2)  0.043(3)  0.034(3)  0.004(2)  -0.005(2)  -0.010(3)  C
C4  0.041(2)  0.051(3)  0.039(3)  0.021(2)  -0.004(2)  -0.016(3)  C
C5  0.024(2)  0.032(2)  0.026(2)  -0.007(2)  0.001(2)  -0.008(2)  C
C6  0.018(2)  0.023(2)  0.023(2)  -0.000(2)  0.003(2)  -0.005(2)  C
C7  0.028(2)  0.033(2)  0.035(2)  0.000(2)  0.000(2)  0.005(2)  C
C8  0.048(2)  0.029(2)  0.038(2)  0.007(3)  0.001(2)  0.011(2)  C
C9  0.044(2)  0.035(3)  0.031(2)  -0.012(2)  0.011(2)  -0.007(2)  C
C10  0.026(2)  0.036(3)  0.034(3)  -0.012(2)  0.009(2)  -0.009(2)  C
C11  0.055(3)  0.015(2)  0.115(5)  -0.002(2)  -0.040(3)  0.001(3)  C
C12  0.040(2)  0.056(3)  0.045(3)  -0.017(2)  0.001(2)  -0.031(3)  C
C13  0.061(3)  0.053(3)  0.039(3)  -0.031(3)  -0.025(2)  0.016(3)  C
C14  0.027(2)  0.045(3)  0.136(6)  -0.012(2)  -0.020(3)  0.010(4)  C
C15  0.070(3)  0.102(4)  0.076(4)  -0.065(3)  0.050(3)  -0.056(3)  C
C16  0.111(4)  0.064(3)  0.047(3)  -0.066(3)  -0.028(3)  0.029(3)  C
P  0.0374(6)  0.0266(6)  0.0234(6)  -0.0045(5)  0.0057(5)  0.0010(6)  P
F1  0.073(2)  0.048(2)  0.073(2)  0.021(2)  0.035(2)  0.005(2)  F
F2  0.053(2)  0.044(2)  0.058(2)  0.014(1)  -0.004(1)  0.012(2)  F
F3  0.038(1)  0.051(2)  0.027(1)  0.005(1)  0.003(1)  0.003(1)  F
F4  0.093(2)  0.071(2)  0.022(1)  -0.039(2)  0.010(1)  -0.003(2)  F
F5  0.045(1)  0.058(2)  0.040(2)  -0.020(1)  0.007(1)  0.001(2)  F
F6  0.064(2)  0.058(2)  0.043(2)  -0.033(1)  0.001(2)  0.001(2)  F

# 10. MOLECULAR GEOMETRY



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RU  N2  2.120(4)  . .  ?
RU  C6  2.064(4)  . .  ?
RU  C11  2.176(5)  . .  ?
RU  C12  2.194(5)  . .  ?
RU  C13  2.167(5)  . .  ?
RU  C14  2.180(5)  . .  ?
RU  C15  2.182(6)  . .  ?
N1  C1  1.139(6)  . .  ?
C1  C2  1.448(7)  . .  ?
N2  C3  1.524(6)  . .  ?
C3  C4  1.513(7)  . .  ?
C3  C5  1.492(7)  . .  ?
C5  C6  1.426(6)  . .  ?
C5  C10  1.372(7)  . .  ?
C6  C7  1.371(6)  . .  ?
C7  C8  1.378(7)  . .  ?
C8  C9  1.366(7)  . .  ?
```

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C9   C10   1.396(7)   .  .   ?
C11  C12   1.37(1)    .  .   ?
C11  C16   1.38(1)    .  .   ?
C12  C13   1.374(9)   .  .   ?
C13  C14   1.33(1)    .  .   ?
C14  C15   1.40(1)    .  .   ?
C15  C16   1.42(1)    .  .   ?
P    F1    1.598(4)   .  .   ?
P    F2    1.585(3)   .  .   ?
P    F3    1.590(3)   .  .   ?
P    F4    1.593(3)   .  .   ?
P    F5    1.593(3)   .  .   ?
P    F6    1.595(3)   .  .   ?

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_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
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N1   RU   N2    85.6(1)    .  .  .   ?
N1   RU   C6    86.3(1)    .  .  .   ?
N1   RU   C11   140.3(3)   .  .  .   ?
N1   RU   C12   166.7(2)   .  .  .   ?
N1   RU   C13   133.1(2)   .  .  .   ?
N1   RU   C14   101.6(3)   .  .  .   ?
N1   RU   C15   88.8(2)    .  .  .   ?
N2   RU   C6    78.2(2)    .  .  .   ?
N2   RU   C11   93.9(2)    .  .  .   ?
N2   RU   C12   106.9(2)   .  .  .   ?
N2   RU   C13   139.1(2)   .  .  .   ?
N2   RU   C14   171.6(2)   .  .  .   ?
N2   RU   C15   139.8(4)   .  .  .   ?
C6   RU   C11   132.6(3)   .  .  .   ?
C6   RU   C12   100.7(2)   .  .  .   ?
C6   RU   C13   90.0(2)    .  .  .   ?
C6   RU   C14   106.4(3)   .  .  .   ?
C6   RU   C15   141.1(4)   .  .  .   ?
C11  RU   C12   36.6(3)    .  .  .   ?
C11  RU   C13   65.8(2)    .  .  .   ?
C11  RU   C14   77.8(2)    .  .  .   ?
C11  RU   C15   66.5(3)    .  .  .   ?
C12  RU   C13   36.7(2)    .  .  .   ?
C12  RU   C14   65.6(3)    .  .  .   ?
C12  RU   C15   78.6(2)    .  .  .   ?
C13  RU   C14   35.7(3)    .  .  .   ?
C13  RU   C15   66.0(3)    .  .  .   ?
C14  RU   C15   37.4(3)    .  .  .   ?
RU   N1   C1    171.2(4)   .  .  .   ?
N1   C1   C2    179.4(5)   .  .  .   ?
RU   N2   C3    113.3(3)   .  .  .   ?
N2   C3   C4    110.3(4)   .  .  .   ?
N2   C3   C5    106.6(4)   .  .  .   ?
C4   C3   C5    113.7(4)   .  .  .   ?
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C3   C5   C6   117.4(4)   .  .   .   ?
C3   C5   C10  121.7(4)   .  .   .   ?
C6   C5   C10  120.9(5)   .  .   .   ?
RU   C6   C5   115.9(3)   .  .   .   ?
RU   C6   C7   127.1(3)   .  .   .   ?
C5   C6   C7   116.9(4)   .  .   .   ?
C6   C7   C8   122.3(4)   .  .   .   ?
C7   C8   C9   120.4(5)   .  .   .   ?
C8   C9   C10  119.5(5)   .  .   .   ?
C5   C10  C9   120.0(4)   .  .   .   ?
RU   C11  C12  72.4(3)    .  .   .   ?
RU   C11  C16  73.8(4)    .  .   .   ?
C12  C11  C16  122.7(6)   .  .   .   ?
RU   C12  C11  71.0(3)    .  .   .   ?
RU   C12  C13  70.5(3)    .  .   .   ?
C11  C12  C13  118.1(6)   .  .   .   ?
RU   C13  C12  72.7(3)    .  .   .   ?
RU   C13  C14  72.7(4)    .  .   .   ?
C12  C13  C14  122.3(6)   .  .   .   ?
RU   C14  C13  71.6(3)    .  .   .   ?
RU   C14  C15  71.4(4)    .  .   .   ?
C13  C14  C15  120.3(7)   .  .   .   ?
RU   C15  C14  71.2(3)    .  .   .   ?
RU   C15  C16  73.1(4)    .  .   .   ?
C14  C15  C16  119.4(7)   .  .   .   ?
C11  C16  C15  117.1(6)   .  .   .   ?
F1   P    F2   178.9(2)   .  .   .   ?
F1   P    F3   88.9(2)    .  .   .   ?
F1   P    F4   90.3(2)    .  .   .   ?
F1   P    F5   89.4(2)    .  .   .   ?
F1   P    F6   89.9(2)    .  .   .   ?
F2   P    F3   90.1(2)    .  .   .   ?
F2   P    F4   90.6(2)    .  .   .   ?
F2   P    F5   90.0(2)    .  .   .   ?
F2   P    F6   90.7(2)    .  .   .   ?
F3   P    F4   179.2(2)   .  .   .   ?
F3   P    F5   90.3(2)    .  .   .   ?
F3   P    F6   89.7(2)    .  .   .   ?
F4   P    F5   89.9(2)    .  .   .   ?
F4   P    F6   90.1(2)    .  .   .   ?
F5   P    F6   179.3(2)   .  .   .   ?


_diffrn_measured_fraction_theta_max    0.998
_diffrn_reflns_theta_full              30.04
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