Understanding conformational preferences of atropisomeric hydrazides and its influence on excited state transformations in crystalline media.

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1. General methods

All commercially obtained reagents/solvents were used as received; chemicals were purchased from Alfa Aesar®, Sigma-Aldrich®, Acros organics®, TCI America®, Mallinckrodt®, and Oakwood® Products, and were used as received without further purification. The synthesis and characterization of hydrazides **1** and **2a-c** as well as their corresponding photoproducts i.e. **3** and **4a-c** are reported in our previous communications.[1] Unless stated otherwise, reactions were conducted in oven-dried glassware under nitrogen atmosphere. The compounds were purified by combiflash equipped with dual wavelength UV-Vis absorbance detector (Teledyne ISCO) using hexanes: ethyl acetate as the mobile phase and Redisep® cartridge filled with silica (Teledyne ISCO) as stationary phase. In some cases, compounds were purified by column chromatography on silica gel (Sorbent Technologies®, silica gel standard grade: porosity 60 Å, particle size: 230 x 400 mesh, surface area: 500 – 600 m2/g, bulk density: 0.4 g/mL, pH range: 6.5 – 7.5). Unless indicated, the Retardation Factor (R_f) values were recorded using a 5-50% hexanes:ethyl acetate as mobile phase and on Sorbent Technologies®, silica Gel TLC plates (200 mm thickness w/UV254).

The residual solvent signal was used as reference. ($CDCl_3$: δ_H = 7.26 ppm, δ_C = 77.2 ppm). Data for ^{1}H NMR spectra are reported as follows: chemical shift (δ ppm), multiplicity, coupling constant (Hz) and integration. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, quin = quintuplet, sext = sextet, sep = septet, b = broad. In some instances, it was not possible to obtain a signal for the carbonyl carbon, despite long relaxation times and concentrated samples. However these signals are reported wherever possible. High-resolution mass spectrum data in Electrospray Ionization mode were recorded on a Bruker – Daltronics® BioTof mass spectrometer in positive (ESI+) ion mode.

HPLC analyses were performed on Waters® HPLC equipped with 2525 pump or on Dionex® Ultimate 3000 HPLC. Waters® 2767 sample manager was used for automated sample injection. All HPLC injections were monitored using a Waters® 2487 dual wavelength absorbance detector at 254 nm and 270 nm. Analytical and semi-preparative injections were performed on chiral stationary phase using various columns indicated below:

i. Regis® PIRKLE COVALENT (R,R) WHELK–01

      a) 25 cm x 4.6 mm column for analytical injections

      b) 25 cm x 10 mm column for semi-preparative injections.

ii. CHIRACEL® OD-H

      a) 0.46 cm x 25 cm column for analytical injections

      b) 25 cm x 10 mm column for semi-preparative injections.

iii. CHIRALPAK® IC

      a) 0.46 cm x 25 cm column for analytical injections

   b) 10 mm x 25 cm column for semi-preparative injections

iv. CHIRALPAK® AD-H

      a) 0.46 cm x 25 cm column for analytical injections

      b) 25 cm x 10 mm column for semi-preparative injections

Masslynx software version 4.1 was used to analyze/process the HPLC injections. Masslynx software version 4.1 was used for analyzing HPLC injections on Waters®. Chromeleon 7.0 software was used for analyzing HPLC injections on Dionex ® HPLC. Igor Pro® Software version 3.0 was used for processing HPLC chromatograms.

1.1. Single crystal XRD analysis

Single crystal X-ray diffraction data of the compounds **2a** and **2c** were collected on a Bruker Apex Duo diffractometer with a Apex 2 CCD area detector at T = 100 K. ImSCu source radiation was used. All structures were process with Apex 2 v2010.9-1 software package (SAINT v. 7.68A, XSHELL v. 6.3.1). Direct method was used to solve the structures after multi-scan absorption corrections.
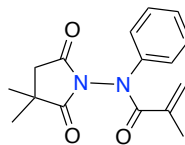
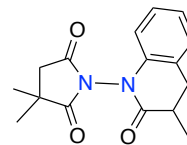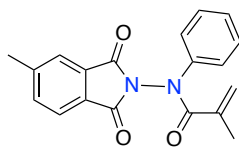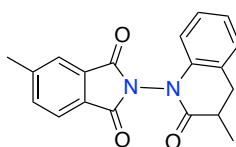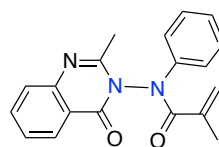## 2. Chemical structures of hydrazides and corresponding photoproducts
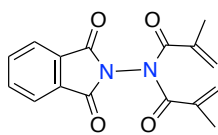
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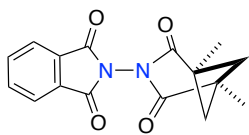
3

2a

4a

2b

4b

2c
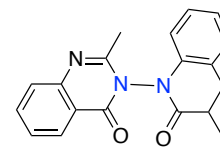
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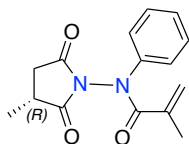
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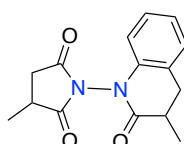
4d

3. Synthetic protocol for the synthesis of hydrazide derivative (R)-2d
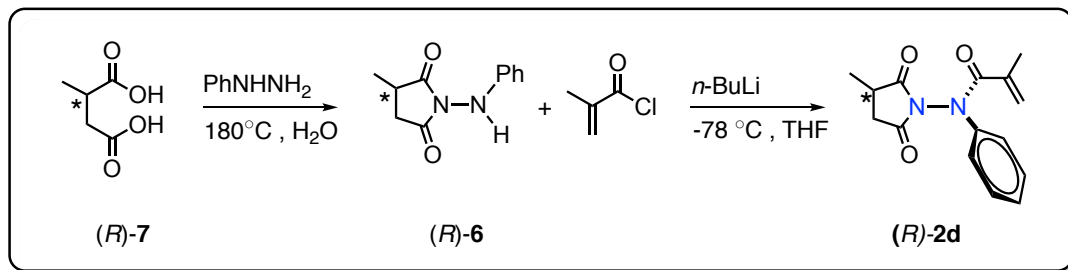
Scheme S1: Synthesis of hydrazide derivative (R)-**2d**.

3.1. Synthesis of hydrazide derivative (R)-6
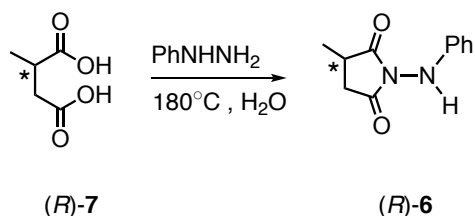
Scheme S2: Synthesis of hydrazide derivative (R)-**6**

Following the modified procedure reported by Kamiński et al., a suspension of *(R)*-(+)-methyl-succinic acid (R)-**7** (1 equiv) in water (10 mL per gram of acid), phenyl hydrazine (1 equiv) was added.[2] The mixture was heated in an oil bath at 180 °C with simultaneous removal of water. After 1 h, the mixture was brought to room temperature and the crude was diluted with ethyl acetate and the organic layer was sequentially washed with 10% HCl, (2 × 10 mL), DI water (2 × 10 mL), saturated $NaHCO_3$ (2 × 10 mL) and finally with brine. The organic layer was dried over anhyd Na_2SO_4, filtered and the solvent was removed under reduced pressure to yield crude product. After concentrating the organic layer, the crude product was purified by combiflash using hexanes and ethyl acetate mixture to get the desired compound.

TLC condition - R_f = 0.3 (50% ethyl acetate:hexanes). Crystalline solid (Yield = 40%)

^{1}H-NMR (400 MHz, CDCl$_3$, δ ppm): 1.41 (d, 3H, *J* 7.2Hz), 2.44 (dd, 1H, J_1 17.6Hz, J_2 3.6Hz), 2.95-3.07 (m, 2H), 6.09 (bs, 1H), 6.76 (d, 2H, *J* 8Hz), 6.97 (t, 1H, *J* 7.6Hz) and 7.21-7.25 (m, 2H).
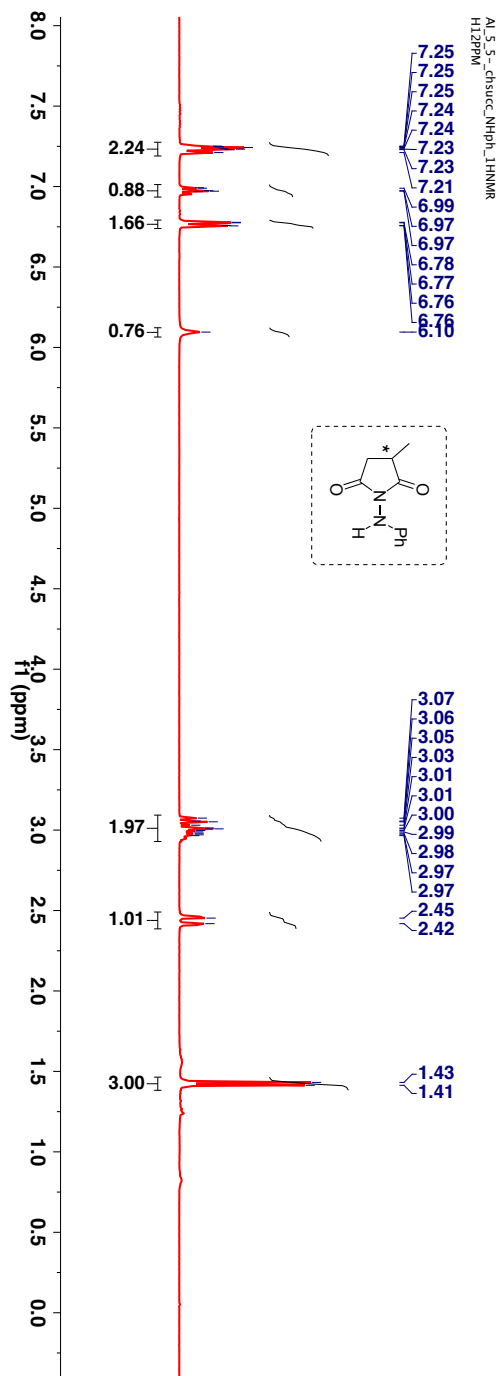
^{13}C-NMR (100 MHz, CDCl$_3$, δ ppm): 17.2, 34.3, 34.9, 114.9, 123.0, 129.6, 145.3, 174.1 and 179.4.
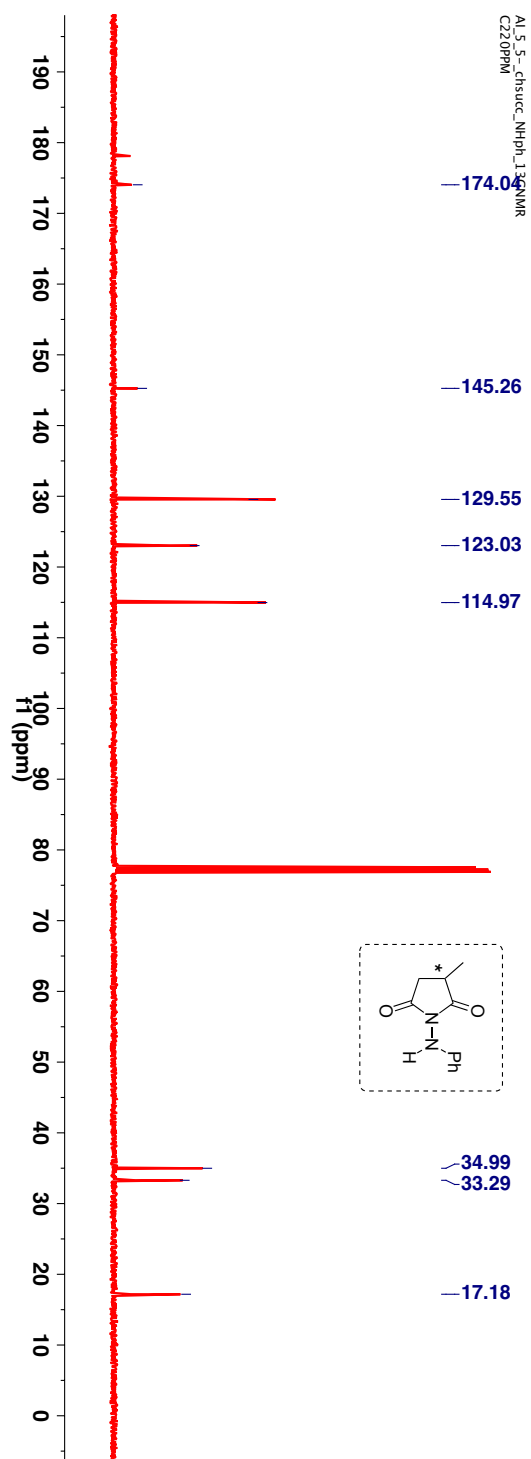
3.2. Synthesis of hydrazide derivative (R)-**2d**
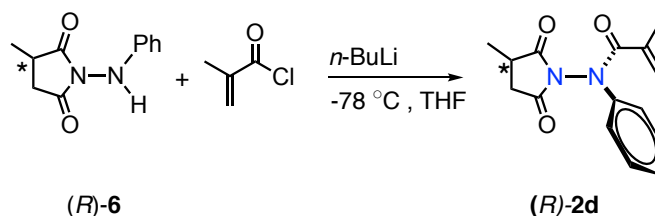
Scheme S3: Synthesis of hydrazide derivative (R)-**2d**

Hydrazide derivative (R)-**6** (1 equiv) was dissolved in anhydrous THF under an inert atmosphere. The solution was then cooled to -78 °C. To this cold solution *n*-BuLi (2.5 M, 1.1 equiv) was added slowly. The mixture was stirred at this temperature for 1 h. The reaction was quenched with anhydrous methacryloyl chloride (1.3 equiv). The stirring was continued for another 2 h after which the solution was quenched with slow addition of ~10 mL of saturated NH_4Cl. (Note: the solution was quenched at -78 °C). The solution was further stirred for 35 min and then diluted with diethyl ether and the organic layer was sequentially washed with DI water (2 × 10 mL), saturated $NaHCO_3$ (2 × 10 mL) and finally with brine. The organic layer was dried over anhyd Na_2SO_4, filtered and the solvent was removed under reduced pressure to yield crude product. After concentrating the organic layer, the crude product was purified by combiflash using hexanes and ethyl acetate mixture to get the desired compound.

TLC condition - R_f = 0.6 (50% ethyl acetate:hexanes).

Crystalline clear solid (Yield = 75%).

Crude NMR

AL_5_45_chiralsucc_crude_1HNMR
H12PPM

7.42
7.42
7.42
7.41
7.40
7.33
7.32
7.32
7.31
7.31
7.31
7.21
7.20
7.20
7.18
6.75
6.75
6.73
6.19
6.18
6.18

5.63
5.63
5.62
5.28
5.28
5.27
5.20

3.05
3.02
3.00
2.99
2.98
2.97
2.96
2.96
2.94
2.90
2.44
2.43
2.39
2.39
2.33
2.30
1.92
1.92
1.82
1.40
1.39
1.31
1.29

*   unreacted acid chloride

∞∞   unreacted starting material
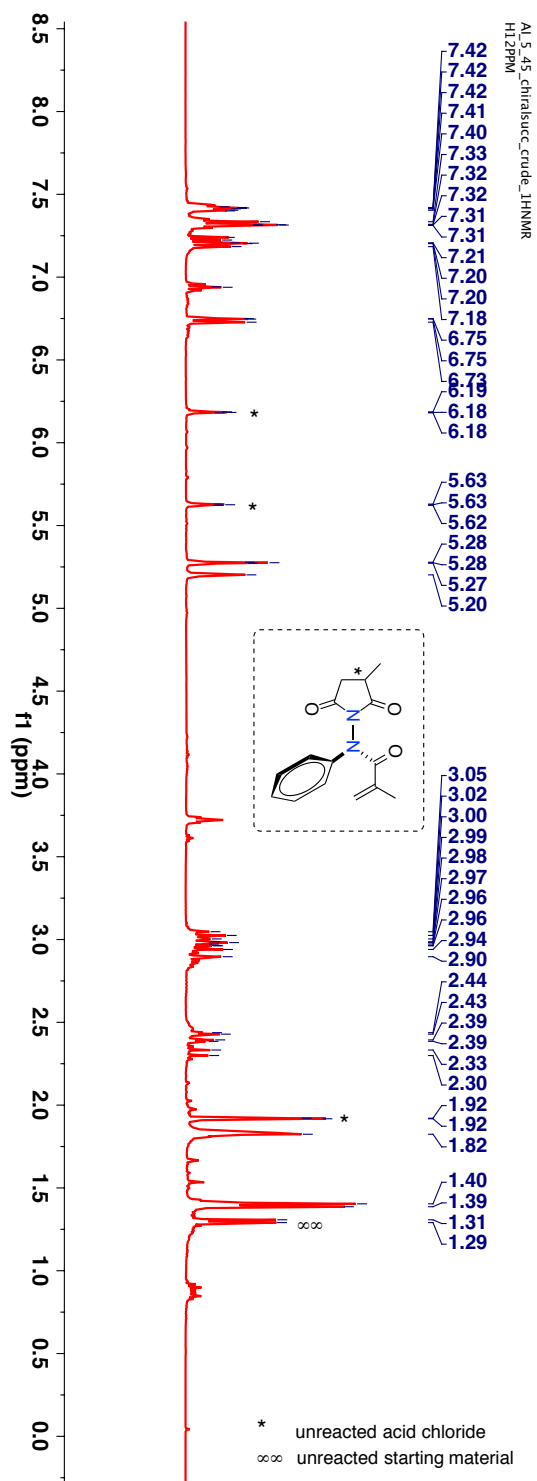
¹H-NMR (400 MHz, CDCl₃, δ ppm, rotamer peaks are reported together): 1.29 (d, *J* 6.8 Hz), 1.39-1.43 (m), 1.83 (bs), 2.28-2.45 (m), 2.84-3.05 (m), 5.19 (s), 5.27 (m), 7.29-7.35 (m) and 7.40-7.43 (m).

Parameters

	Parameter	Value (f2, f1)
1	Origin	Bruker BioSpin GmbH
2	Solvent	CDCl3
3	Temperature	298.2
4	Experiment	COSY
5	Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0645
6	Number of Scans	4
7	Receiver Gain	197
8	Relaxation Delay	2.0000
9	Pulse Width	14.7000
10	Acquisition Date	2015-07-29T12:52:02
11	Spectrometer Frequency	(400.13, 400.13)
12	Spectral Width	(5341.9, 5341.9)
13	Lowest Frequency	(-265.3, -265.3)
14	Nucleus	(1H, 1H)
15	Acquired Size	(1024, 128)
16	Spectral Size	(1024, 1024)

Parameters

	Parameter	Value (f2, f1)
1	Origin	Bruker BioSpin GmbH
2	Solvent	CDCl3
3	Temperature	298.2
4	Experiment	COSY
5	Probe	5 mm PABBO BB/ 19F-1H/ D Z-GRD Z108618/ 0645
6	Number of Scans	4
7	Receiver Gain	197
8	Relaxation Delay	2.0000
9	Pulse Width	14.7000
10	Acquisition Date	2015-07-29T12:52:02
11	Spectrometer Frequency	(400.13, 400.13)
12	Spectral Width	(5341.9, 5341.9)
13	Lowest Frequency	(-265.3, -265.3)
14	Nucleus	(1H, 1H)
15	Acquired Size	(1024, 128)
16	Spectral Size	(1024, 1024)

Figure S1: COSY (400 MHz, CDCl$_3$, δ ppm) spectrum of (*R*)-**2d**.

Figure S2: NOESY (400 MHz, CDCl₃, δ ppm) spectrum of (*R*)-**2d**.

^{13}C-NMR (100 MHz, CDCl$_3$, δ ppm, rotamer peaks are reported together): 21.7, 22.0, 22.1, 38.4, 38.5, 39.9, 40.0, 41.9, 131.6, 132.7, 132.9, 133.8, 133.9, 133.9, 134.3, 134.5, 134.6, 177.5, 177.7, 181.6 and 181.7.
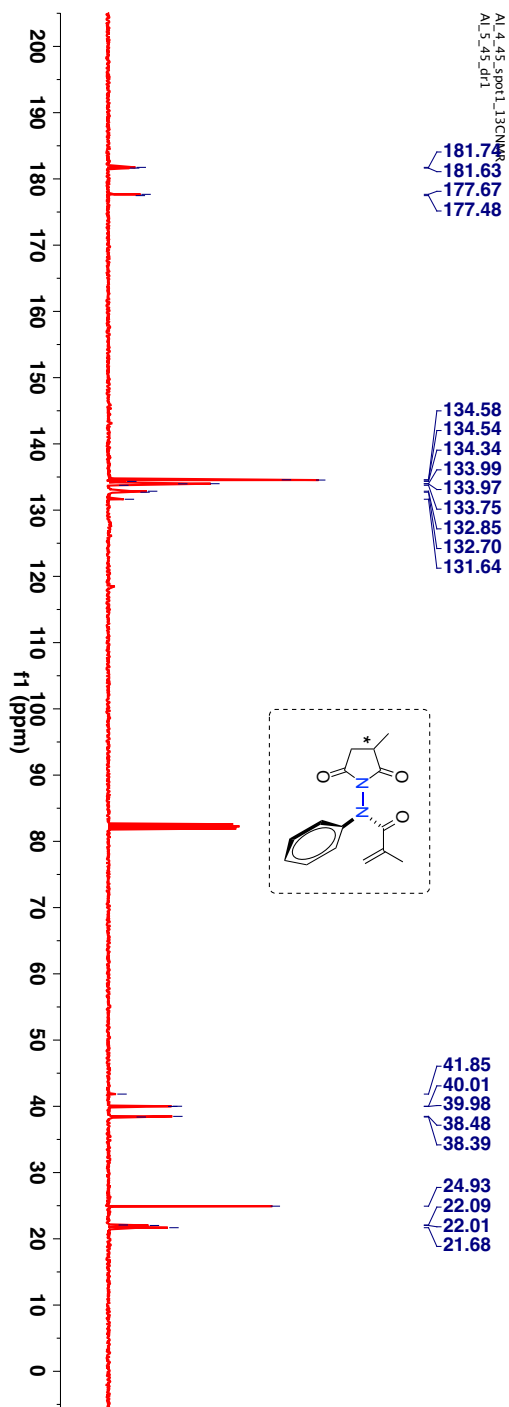
HRMS-ESI (m/z) ([M + Na]):

Chemical Formula: $C_{16}H_{20}N_2O_3$

Calculated   : 295.1059

Observed   : 295.1073

|Δm| : 4.7 ppm
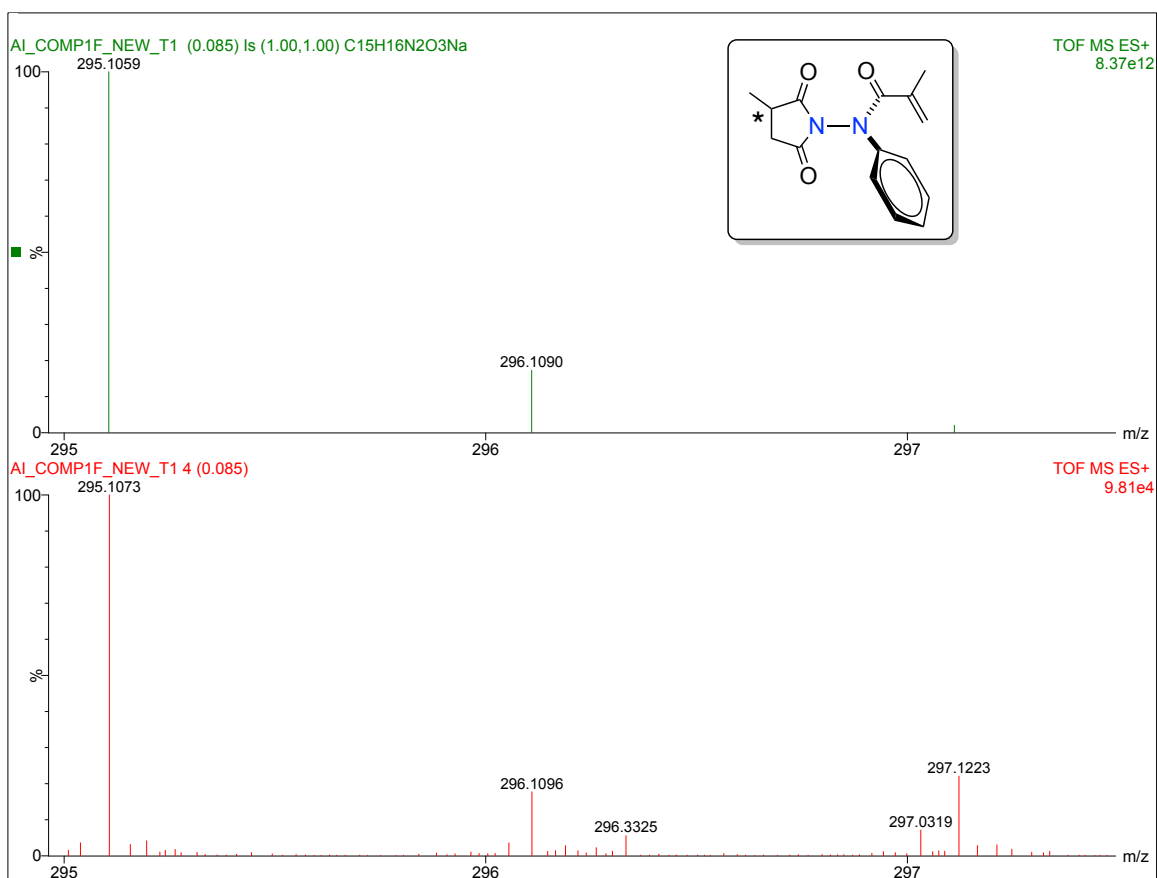
4. General irradiation procedures and characterization of photoproduct(s)

4.1. Procedure for photoreaction

a) Sensitized irradiation

In a pyrex tube, solution of the desired substrate was dissolved in an appropriate solvent. This was followed by addition of thioxanthone and then the solution was deoxygenated by purging N_2. The sample was then irradiated at ambient temperature in a Rayonet reactor equipped with ~420 nm tubes (16 tubes x 14 Watt).

b) Direct irradiation

In a pyrex tube, solution of the desired substrate was dissolved in an appropriate solvent. The solution was then deoxygenated by purging N_2. The sample was then irradiated at ambient temperature in a Rayonet reactor equipped with ~300 nm tubes (16 tubes x 12 Watt).

5. Photoreaction of hydrazide derivative (*R*)-2d in solution

(*R*)-**2d** hν λ ~ 300 nm, Acetonitrile, 4 h, N$_2$ → (*R*)-**4d**
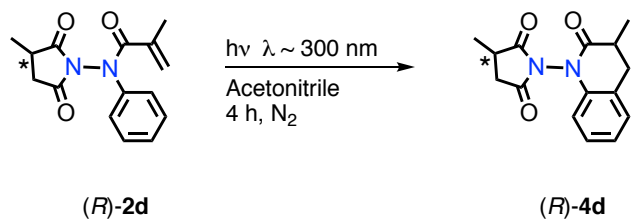
Scheme S4: 6π Photocyclization of hydrazide derivative (*R*)-**2d** by direct irradiation.

(*R*)-**2d** hν λ ~ 300 nm, 10 mol% xanthone, Acetonitrile, 4 h, N$_2$ → (*R*)-**4d**
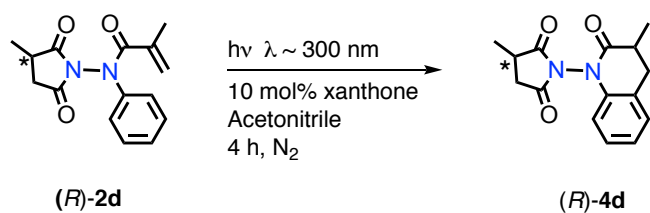
Scheme S5: 6π Photocyclization of hydrazide derivative (*R*)-**2d** under the conditions of sensitized irradiation.

5.1. Characterization of photoproduct (*R*)-**4d**

TLC condition - R_f = 0.4 (50% hexanes:ethyl acetate). Clear solid (Yield = 75%).

^{1}H-NMR (400 MHz, CDCl$_3$, δ ppm): 1.31-1.33 (m), 1.46-1.52 (m), 2.52-2.61(m), 2.90-3.18 (m), 6.53-6.61 (m), 7.01-7.05 (m) and 7.14-7.20 (m).
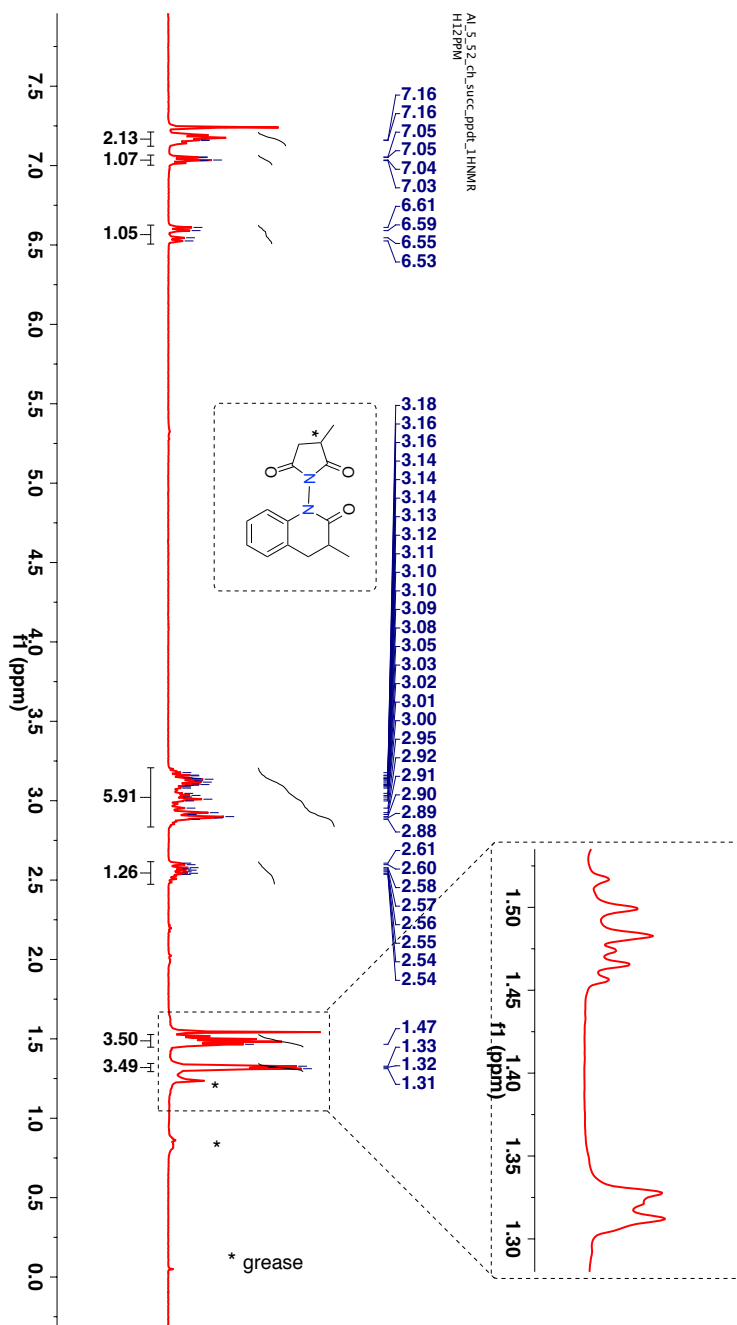
^{13}C-NMR (100 MHz, CDCl$_3$, δ ppm): 15.4, 15.4, 17.1, 17.3, 17.4, 17.4, 33.1, 33.2, 33.6, 33.7, 33.8, 35.1, 35.2, 35.2, 35.3, 35.7, 35.7, 112.6, 112.7, 112.9, 112.9, 124.4, 124.7, 124.7, 127.9, 127.9, 127.9, 128.5, 128.5, 128.6, 138.1, 169.6, 169.6, 172.1, 172.7, 175.9, 176.2 and 176.8.
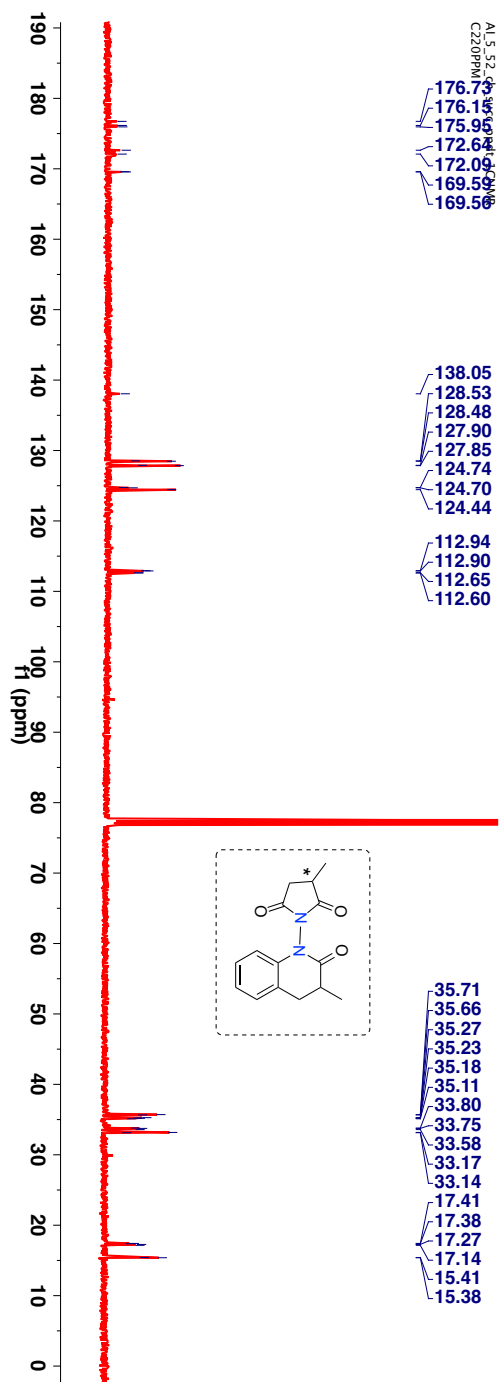
6. HPLC analysis for hydrazides

6.1. HPLC analysis for hydrazide derivative (R)-**6**
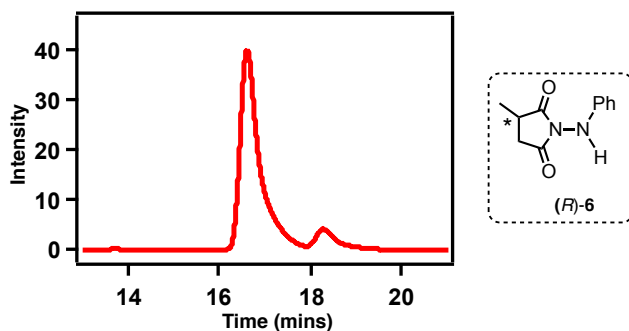
For analytical conditions,

Column	: CHIRALPAK® AD-H
Abs. detector wavelength	: 254 nm and 270 nm
Mobile phase	: Hexanes:2-propanol = 80:20
Flow rate	: 0.8 mL/min
Retention times (min)	: pA 16.6 and pB 18.3
Relative area	: pA ~ 94% and pB ~ 6%

6.2. HPLC analysis for hydrazide derivative (R)-**2d**
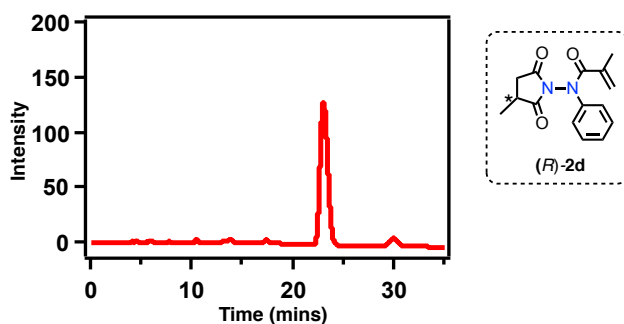
Column	: CHIRALPAK® IC
Abs. detector wavelength	: 254 nm and 270 nm
Mobile phase	: Hexanes:2-propanol = 70:30
Flow rate	: 0.8 mL/min
Retention times (min)	: pA ~23.0

6.3. HPLC analysis conditions for hydrazide derivative (*R*)-**4d** by direct irradiation (crude sample was injected)
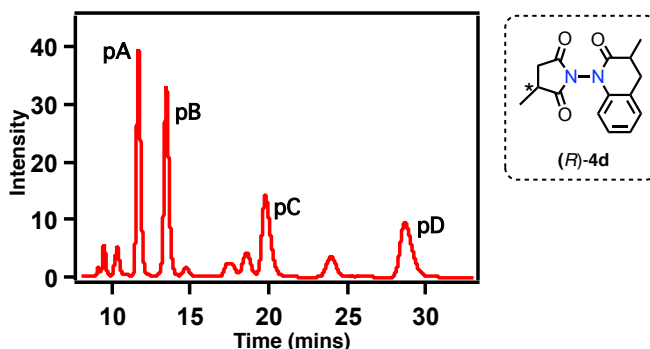
For analytical conditions,

Column	: CHIRALPAK® IC
Abs. detector wavelength	: 254 nm and 270 nm
Mobile phase	: Hexanes:2-propanol = 70:30
Flow rate	: 0.8 mL/min
Retention times (min)	: pA 11.6, pB 13.4, pC 19.7 and 28.6
Relative area	: pA ~ 29%, pB ~ 29%, pC = 21%, pD ~ 21%

6.4. HPLC analysis conditions for hydrazide derivative (*R*)-**4d** by sensitized irradiation (crude sample was injected)
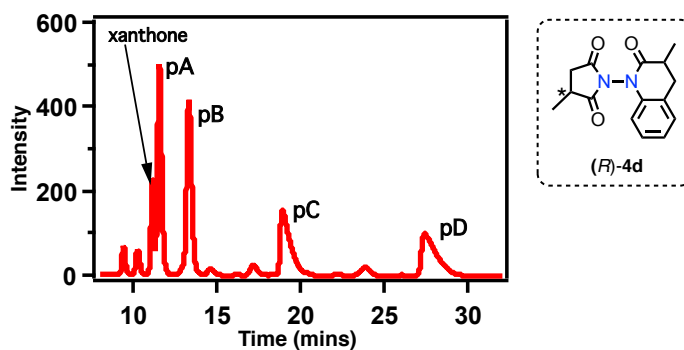
Column	: CHIRALPAK® IC
Abs. detector wavelength	: 254 nm and 270 nm
Mobile phase	: Hexanes:2-propanol = 70:30
Flow rate	: 0.8 mL/min
Retention times (min)	: pA 11.6, pB 13.4, pC 19.7 and 28.6
Relative area	: pA ~ 29%, pB ~ 29%, pC ~ 21%, pD ~ 21%

7. Racemization kinetics of hydrazide derivative 2c

N-N bond rotation

$\Delta G^{\ddagger}_{rac} \sim 24$ kcal.mol^{-1}

$T = 45\ °C$

2c-enantiomer1   **2c-enantiomer2**

ethylacetate, 45 ºC
k_{rac} (s^{-1}) = 16.4 x 10^{-5}
$\tau_{1/2}$ (h) = 1.2
$\Delta G^{\ddagger}_{rac}$(kcal.mol^{-1}) = 24.2

ethylacetate, 45 ºC
k_{rac} (s^{-1}) = 16.4 x 10^{-5}
$\tau_{1/2}$ (h) = 1.2
$\Delta G^{\ddagger}_{rac}$(kcal.mol^{-1}) = 24.2

Benzene, 45 ºC
k_{rac} (s^{-1}) = 2.15 x 10^{-4}
$\tau_{1/2}$ (h) = 0.89
$\Delta G^{\ddagger}_{rac}$(kcal.mol^{-1}) = 23.9

Benzene, 45 ºC
k_{rac} (s^{-1}) = 2.15 x 10^{-4}
$\tau_{1/2}$ (h) = 0.89
$\Delta G^{\ddagger}_{rac}$(kcal.mol^{-1}) = 23.9
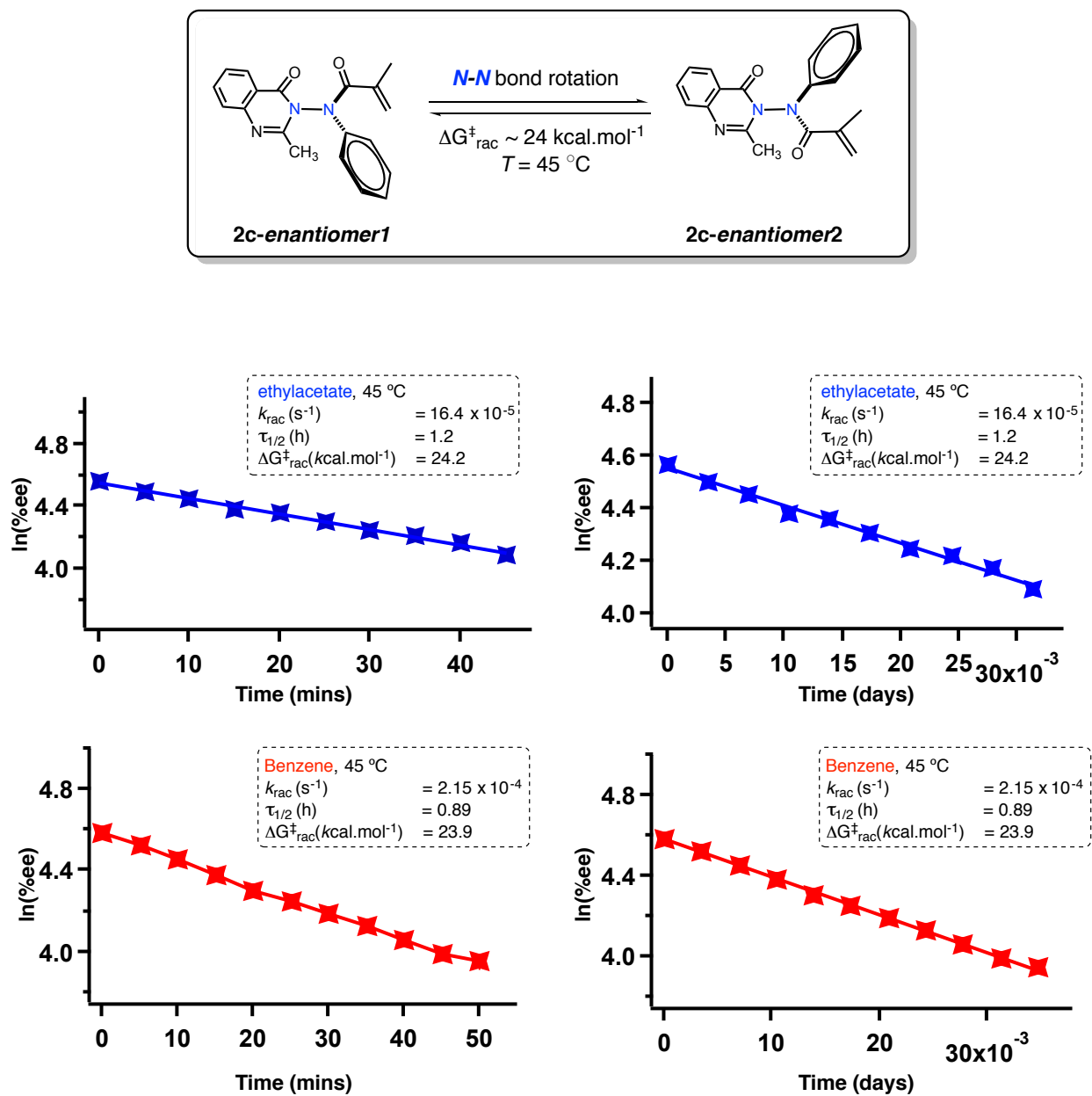
Figure S3: Racemization kinetics for quinazolinone based *N-N* atropisomer **2c** in benzene and ethyl acetate.

8. References

[1]   a) A. Iyer, S. Jockusch, J. Sivaguru, *Chem Commun* **2017**, *53(10)*, 1692-1695; b) A. Iyer, S. Ahuja, S. Jockusch, A. Ugrinov, J. Sivaguru, *Chem Commun* **2018**, *54(78)*, 1021-11024; c) A. Iyer, North Dakota State Univeristy **2016**.

[2]   K. Kamiński, J. Obniska, *Bioorganic & Medicinal Chemistry* **2008**, *16*, 4921-4931.

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_exptl_crystal_density_meas        ?
_exptl_crystal_density_method      ?
_exptl_crystal_description         'chunk'
_exptl_crystal_F_000               1152
_exptl_crystal_size_max            0.24
_exptl_crystal_size_mid            0.174
_exptl_crystal_size_min            0.148
_exptl_transmission_factor_max     ?
_exptl_transmission_factor_min     ?
_diffrn_reflns_av_R_equivalents    0.0339
_diffrn_reflns_av_unetI/netI       0.0638
_diffrn_reflns_Laue_measured_fraction_full 0.974
_diffrn_reflns_Laue_measured_fraction_max 0.974
_diffrn_reflns_limit_h_max         10
```

```
_diffrn_reflns_limit_h_min            -10
_diffrn_reflns_limit_k_max            27
_diffrn_reflns_limit_k_min            -28
_diffrn_reflns_limit_l_max            16
_diffrn_reflns_limit_l_min            -16
_diffrn_reflns_number                 17719
_diffrn_reflns_point_group_measured_fraction_full 0.871
_diffrn_reflns_point_group_measured_fraction_max 0.870
_diffrn_reflns_theta_full             66.779
_diffrn_reflns_theta_max              66.779
_diffrn_reflns_theta_min              3.723
_diffrn_ambient_temperature           100.01
_diffrn_detector_area_resol_mean      ?
_diffrn_measured_fraction_theta_full 0.974
_diffrn_measured_fraction_theta_max 0.974
_diffrn_measurement_device_type       'Bruker APEX-II CCD'
_diffrn_measurement_method            '\f and \w scans'
_diffrn_radiation_type                CuK\a
_diffrn_radiation_wavelength          1.54178
_diffrn_source                        ?
_diffrn_standards_number              0
_reflns_Friedel_coverage              0.741
_reflns_Friedel_fraction_full         0.761
_reflns_Friedel_fraction_max          0.761
_reflns_number_gt                     7549
_reflns_number_total                  8826
_reflns_special_details
;
 Reflections were merged by SHELXL according to the crystal
 class for the calculation of statistics and refinement.

 _reflns_Friedel_fraction is defined as the number of unique
 Friedel pairs measured divided by the number that would be
 possible theoretically, ignoring centric projections and
 systematic absences.
;
_reflns_threshold_expression          'I > 2\s(I)'
_computing_cell_refinement            'SAINT v8.34A (Bruker, 2013)'
_computing_data_collection            ?
_computing_data_reduction             'SAINT v8.34A (Bruker, 2013)'
_computing_molecular_graphics         'Olex2 (Dolomanov et al., 2009)'
_computing_publication_material       'Olex2 (Dolomanov et al., 2009)'
_computing_structure_refinement       'XL (Sheldrick, 2008)'
_computing_structure_solution         ?
_refine_diff_density_max              0.503
_refine_diff_density_min              -0.294
_refine_diff_density_rms              0.052
_refine_ls_abs_structure_details
;
 Flack x determined using 2667 quotients [(I+)-(I-)]/[(I+)+(I-)]
```

(Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249–259).
;
_refine_ls_abs_structure_Flack   −0.1(3)
_refine_ls_extinction_coef   .
_refine_ls_extinction_method   none
_refine_ls_goodness_of_fit_ref   1.041
_refine_ls_hydrogen_treatment   constr
_refine_ls_matrix_type   full
_refine_ls_number_parameters   749
_refine_ls_number_reflns   8826
_refine_ls_number_restraints   1
_refine_ls_R_factor_all   0.0700
_refine_ls_R_factor_gt   0.0610
_refine_ls_restrained_S_all   1.041
_refine_ls_shift/su_max   0.000
_refine_ls_shift/su_mean   0.000
_refine_ls_structure_factor_coef   Fsqd
_refine_ls_weighting_details
'w=1/[\s^2^(Fo^2^)+(0.0730P)^2^+1.9154P] where P=(Fo^2^+2Fc^2^)/3'
_refine_ls_weighting_scheme   calc
_refine_ls_wR_factor_gt   0.1597
_refine_ls_wR_factor_ref   0.1668
_refine_special_details   ?
_olex2_refinement_description
;
1. Fixed Uiso
 At 1.2 times of:
  All C(H) groups, {H12D,H12A,H12B} of C12D, All C(H,H) groups
 At 1.5 times of:
  {H1CA,H1CB,H1CC} of C1C, {H16A,H16B,H16C} of C16D, {H1BA,H1BB,H1BC}
of C1B,
  {H1DA,H1DB,H1DC} of C1D, {H15G,H15H,H15I} of C15A, {H1AA,H1AB,H1AC}
of C1A,
  {H15D,H15E,H15F} of C15B, {H15A,H15B,H15C} of C15D, {H15J,H15K,H15L}
of C15C
2. Others

Sof(H12D)=Sof(C13D)=Sof(H13A)=Sof(H13B)=Sof(C15D)=Sof(H15A)=Sof(H15B)=
 Sof(H15C)=1−FVAR(1)

Sof(H12A)=Sof(H12B)=Sof(C16D)=Sof(H16A)=Sof(H16B)=Sof(H16C)=Sof(C17D)=
 Sof(H17D)=FVAR(1)
3.a Ternary CH refined with riding coordinates:
 C12D(H12D), C17D(H17D), C13B(H13C), C13A(H13D), C13C(H13E)
3.b Secondary CH2 refined with riding coordinates:
 C12D(H12A,H12B), C13D(H13A,H13B), C12B(H12C,H12E), C12A(H12F,H12G),
C12C(H12H,
 H12I)
3.c Aromatic/amide H refined with riding coordinates:
 C6D(H6D), C7D(H7D), C8D(H8D), C9D(H9D), C10D(H10D), C6B(H6B),

C7B(H7B),
 C8B(H8B), C9B(H9B), C10B(H10B), C6A(H6A), C7A(H7A), C8A(H8A),
C9A(H9A),
 C10A(H10A), C6C(H6C), C7C(H7C), C8C(H8C), C9C(H9C), C10C(H10C)
3.d X=CH2 refined with riding coordinates:
 C3D(H3DA,H3DB), C3B(H3BA,H3BB), C3A(H3AA,H3AB), C3C(H3CA,H3CB)
3.e Idealised Me refined as rotating group:
 C1D(H1DA,H1DB,H1DC), C15D(H15A,H15B,H15C), C16D(H16A,H16B,H16C),
C1B(H1BA,
 H1BB,H1BC), C15B(H15D,H15E,H15F), C1A(H1AA,H1AB,H1AC),
C15A(H15G,H15H,H15I),
 C1C(H1CA,H1CB,H1CC), C15C(H15J,H15K,H15L)
;
_atom_sites_solution_hydrogens   geom
_atom_sites_solution_primary   ?
_atom_sites_solution_secondary   ?
loop_
  _atom_site_label
  _atom_site_type_symbol
  _atom_site_fract_x
  _atom_site_fract_y
  _atom_site_fract_z
  _atom_site_U_iso_or_equiv
  _atom_site_adp_type
  _atom_site_occupancy
  _atom_site_site_symmetry_order
  _atom_site_calc_flag
  _atom_site_refinement_flags_posn
  _atom_site_refinement_flags_adp
  _atom_site_refinement_flags_occupancy
  _atom_site_disorder_assembly
  _atom_site_disorder_group
C1D C 2.7361(9) 1.7499(3) 1.0756(5) 0.0486(16) Uani 1 1 d . . . . . .
H1DA H 2.6577 1.7519 1.0133 0.073 Uiso 1 1 calc GR . . . .
H1DB H 2.8385 1.7609 1.0661 0.073 Uiso 1 1 calc GR . . . .
H1DC H 2.7066 1.7757 1.1224 0.073 Uiso 1 1 calc GR . . . .
C2D C 2.7442(6) 1.6905(2) 1.1142(4) 0.0296(11) Uani 1 1 d . . . . . .
C3D C 2.6659(7) 1.6469(3) 1.0646(4) 0.0361(13) Uani 1 1 d . . . . . .
H3DA H 2.6819 1.6098 1.0907 0.043 Uiso 1 1 calc R . . . .
H3DB H 2.5944 1.6531 1.0034 0.043 Uiso 1 1 calc R . . . .
C4D C 2.8566(6) 1.6819(2) 1.2100(4) 0.0279(11) Uani 1 1 d . . . . . .
C5D C 2.6442(6) 1.6388(2) 1.2799(3) 0.0256(11) Uani 1 1 d . . . . . .
C6D C 2.5318(6) 1.6789(3) 1.2851(4) 0.0335(12) Uani 1 1 d . . . . . .
H6D H 2.5568 1.7180 1.2904 0.040 Uiso 1 1 calc R . . . .
C7D C 2.3816(7) 1.6602(3) 1.2822(4) 0.0428(15) Uani 1 1 d . . . . . .
H7D H 2.3025 1.6868 1.2858 0.051 Uiso 1 1 calc R . . . .
C8D C 2.3455(7) 1.6033(3) 1.2740(4) 0.0470(17) Uani 1 1 d . . . . . .
H8D H 2.2416 1.5910 1.2706 0.056 Uiso 1 1 calc R . . . .
C9D C 2.4600(7) 1.5644(3) 1.2709(5) 0.0430(15) Uani 1 1 d . . . . . .
H9D H 2.4359 1.5252 1.2677 0.052 Uiso 1 1 calc R . . . .

```
C10D C 2.6103(7) 1.5820(3) 1.2725(4) 0.0332(12) Uani 1 1 d . . . . . .
H10D H 2.6889 1.5553 1.2685 0.040 Uiso 1 1 calc R . . . .
C11D C 2.9535(7) 1.6895(3) 1.4414(4) 0.0398(14) Uani 1 1 d . . . . . .
C12D C 3.0865(7) 1.6635(3) 1.5187(4) 0.0491(17) Uani 1 1 d . . . . . .
H12D H 3.0566 1.6663 1.5823 0.059 Uiso 0.452(17) 1 calc R . P A 2
H12A H 3.0473 1.6483 1.5735 0.059 Uiso 0.548(17) 1 calc R . P A 1
H12B H 3.1688 1.6919 1.5443 0.059 Uiso 0.548(17) 1 calc R . P A 1
C13D C 3.103(2) 1.6047(10) 1.4997(15) 0.061(6) Uani 0.452(17) 1 d . .
P A 2
H13A H 3.0524 1.5807 1.5406 0.074 Uiso 0.452(17) 1 calc R . P A 2
H13B H 3.2146 1.5938 1.5107 0.074 Uiso 0.452(17) 1 calc R . P A 2
C14D C 3.0121(7) 1.6005(4) 1.3844(6) 0.059(2) Uani 1 1 d . . . . . .
C15D C 3.2279(19) 1.6955(8) 1.5282(12) 0.064(5) Uani 0.452(17) 1
d . . P A 2
H15A H 3.2530 1.6982 1.4645 0.097 Uiso 0.452(17) 1 calc GR . P A 2
H15B H 3.3139 1.6766 1.5744 0.097 Uiso 0.452(17) 1 calc GR . P A 2
H15C H 3.2134 1.7335 1.5520 0.097 Uiso 0.452(17) 1 calc GR . P A 2
C16D C 3.1995(17) 1.5618(6) 1.5239(10) 0.066(5) Uani 0.548(17) 1
d . . P A 1
H16A H 3.2902 1.5701 1.5778 0.099 Uiso 0.548(17) 1 calc GR . P A 1
H16B H 3.2270 1.5322 1.4826 0.099 Uiso 0.548(17) 1 calc GR . P A 1
H16C H 3.1123 1.5489 1.5502 0.099 Uiso 0.548(17) 1 calc GR . P A 1
C17D C 3.1519(14) 1.6145(6) 1.4642(9) 0.038(3) Uani 0.548(17) 1 d . .
P A 1
H17D H 3.2396 1.6285 1.4369 0.046 Uiso 0.548(17) 1 calc R . P A 1
N1D N 2.8019(5) 1.6566(2) 1.2823(3) 0.0307(10) Uani 1 1 d . . . . . .
N2D N 2.9119(5) 1.6475(2) 1.3701(3) 0.0338(11) Uani 1 1 d . . . . . .
O1D O 2.9941(4) 1.69657(18) 1.2242(3) 0.0381(9) Uani 1 1 d . . . . . .
O2D O 3.0003(5) 1.5614(2) 1.3287(5) 0.0704(17) Uani 1 1 d . . . . . .
O3D O 2.8875(6) 1.7336(2) 1.4375(4) 0.0526(12) Uani 1 1 d . . . . . .
C1B C 3.2275(8) 1.7749(3) 1.0843(4) 0.0400(14) Uani 1 1 d . . . . . .
H1BA H 3.1617 1.7742 1.0176 0.060 Uiso 1 1 calc GR . . . .
H1BB H 3.1844 1.7489 1.1250 0.060 Uiso 1 1 calc GR . . . .
H1BC H 3.3341 1.7632 1.0840 0.060 Uiso 1 1 calc GR . . . .
C2B C 3.2308(6) 1.8335(2) 1.1244(4) 0.0310(12) Uani 1 1 d . . . . . .
C3B C 3.1518(7) 1.8768(3) 1.0777(4) 0.0350(13) Uani 1 1 d . . . . . .
H3BA H 3.0872 1.8719 1.0138 0.042 Uiso 1 1 calc R . . . .
H3BB H 3.1597 1.9129 1.1079 0.042 Uiso 1 1 calc R . . . .
C4B C 3.3450(6) 1.8397(3) 1.2216(4) 0.0334(12) Uani 1 1 d . . . . . .
C5B C 3.1302(6) 1.8799(3) 1.2922(4) 0.0318(12) Uani 1 1 d . . . . . .
C6B C 3.0207(7) 1.8389(3) 1.2973(4) 0.0369(13) Uani 1 1 d . . . . . .
H6B H 3.0500 1.8002 1.3041 0.044 Uiso 1 1 calc R . . . .
C7B C 2.8676(7) 1.8549(3) 1.2925(5) 0.0450(16) Uani 1 1 d . . . . . .
H7B H 2.7910 1.8271 1.2953 0.054 Uiso 1 1 calc R . . . .
C8B C 2.8267(7) 1.9112(3) 1.2838(5) 0.0442(16) Uani 1 1 d . . . . . .
H8B H 2.7216 1.9219 1.2803 0.053 Uiso 1 1 calc R . . . .
C9B C 2.9363(7) 1.9521(3) 1.2800(4) 0.0380(13) Uani 1 1 d . . . . . .
H9B H 2.9066 1.9907 1.2746 0.046 Uiso 1 1 calc R . . . .
C10B C 3.0921(6) 1.9368(2) 1.2841(4) 0.0318(12) Uani 1 1 d . . . . . .
H10B H 3.1688 1.9646 1.2814 0.038 Uiso 1 1 calc R . . . .
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C11B C 3.4287(11) 1.8183(5) 1.4447(6) 0.083(3) Uani 1 1 d . . . . . .
C12B C 3.5646(14) 1.8318(6) 1.5279(8) 0.119(5) Uani 1 1 d . . . . . .
H12C H 3.6567 1.8085 1.5251 0.143 Uiso 1 1 calc R . . . .
H12E H 3.5379 1.8251 1.5910 0.143 Uiso 1 1 calc R . . . .
C13B C 3.5964(10) 1.8937(7) 1.5148(8) 0.112(5) Uani 1 1 d . . . . . .
H13C H 3.5621 1.9158 1.5661 0.134 Uiso 1 1 calc R . . . .
C14B C 3.4947(9) 1.9109(5) 1.4142(7) 0.071(3) Uani 1 1 d . . . . . .
C15B C 3.7523(16) 1.9046(11) 1.5219(10) 0.201(10) Uani 1 1
d . . . . . .
H15D H 3.7660 1.9449 1.5097 0.301 Uiso 1 1 calc GR . . . .
H15E H 3.7884 1.8820 1.4736 0.301 Uiso 1 1 calc GR . . . .
H15F H 3.8135 1.8948 1.5874 0.301 Uiso 1 1 calc GR . . . .
N1B N 3.2894(5) 1.8638(2) 1.2951(3) 0.0381(12) Uani 1 1 d . . . . . .
N2B N 3.3956(6) 1.8664(3) 1.3855(4) 0.0508(15) Uani 1 1 d . . . . . .
O1B O 3.4801(5) 1.82406(19) 1.2360(3) 0.0441(10) Uani 1 1 d . . . . . .
O2B O 3.4936(6) 1.9529(3) 1.3697(5) 0.0776(19) Uani 1 1 d . . . . . .
O3B O 3.3596(10) 1.7735(3) 1.4285(5) 0.102(3) Uani 1 1 d . . . . . .
C1A C 2.6771(11) 1.5714(4) 1.5403(5) 0.074(3) Uani 1 1 d . . . . . .
H1AA H 2.6247 1.5836 1.4742 0.111 Uiso 1 1 calc GR . . . .
H1AB H 2.6096 1.5448 1.5641 0.111 Uiso 1 1 calc GR . . . .
H1AC H 2.7762 1.5528 1.5394 0.111 Uiso 1 1 calc GR . . . .
C2A C 2.7088(7) 1.6218(3) 1.6063(4) 0.0444(16) Uani 1 1 d . . . . . .
C3A C 2.6654(10) 1.6735(4) 1.5807(6) 0.068(2) Uani 1 1 d . . . . . .
H3AA H 2.6091 1.6813 1.5158 0.082 Uiso 1 1 calc R . . . .
H3AB H 2.6901 1.7032 1.6270 0.082 Uiso 1 1 calc R . . . .
C4A C 2.8133(7) 1.6087(3) 1.7048(4) 0.0353(13) Uani 1 1 d . . . . . .
C5A C 2.5925(6) 1.6340(2) 1.7811(3) 0.0270(11) Uani 1 1 d . . . . . .
C6A C 2.4844(7) 1.5912(3) 1.7606(4) 0.0388(13) Uani 1 1 d . . . . . .
H6A H 2.5145 1.5540 1.7474 0.047 Uiso 1 1 calc R . . . .
C7A C 2.3296(7) 1.6035(3) 1.7595(5) 0.0473(17) Uani 1 1 d . . . . . .
H7A H 2.2533 1.5744 1.7451 0.057 Uiso 1 1 calc R . . . .
C8A C 2.2858(7) 1.6562(3) 1.7786(4) 0.0467(17) Uani 1 1 d . . . . . .
H8A H 2.1797 1.6636 1.7781 0.056 Uiso 1 1 calc R . . . .
C9A C 2.3936(8) 1.6986(3) 1.7986(5) 0.0536(18) Uani 1 1 d . . . . . .
H9A H 2.3621 1.7356 1.8118 0.064 Uiso 1 1 calc R . . . .
C10A C 2.5482(7) 1.6883(3) 1.7998(5) 0.0408(14) Uani 1 1 d . . . . . .
H10A H 2.6231 1.7179 1.8133 0.049 Uiso 1 1 calc R . . . .
C11A C 2.8717(7) 1.5604(3) 1.9206(5) 0.0375(13) Uani 1 1 d . . . . . .
C12A C 2.9951(8) 1.5662(3) 2.0153(5) 0.0501(17) Uani 1 1 d . . . . . .
H12F H 3.0792 1.5379 2.0194 0.060 Uiso 1 1 calc R . . . .
H12G H 2.9486 1.5612 2.0719 0.060 Uiso 1 1 calc R . . . .
C13A C 3.0591(6) 1.6266(3) 2.0126(4) 0.0375(13) Uani 1 1 d . . . . . .
H13D H 3.0449 1.6480 2.0708 0.045 Uiso 1 1 calc R . . . .
C14A C 2.9612(6) 1.6540(3) 1.9213(4) 0.0333(13) Uani 1 1 d . . . . . .
C15A C 3.2315(7) 1.6270(3) 2.0119(5) 0.0497(15) Uani 1 1 d . . . . . .
H15G H 3.2478 1.6030 1.9589 0.075 Uiso 1 1 calc GR . . . .
H15H H 3.2939 1.6127 2.0743 0.075 Uiso 1 1 calc GR . . . .
H15I H 3.2640 1.6658 2.0021 0.075 Uiso 1 1 calc GR . . . .
N1A N 2.7544(5) 1.6229(2) 1.7838(3) 0.0282(10) Uani 1 1 d . . . . . .
N2A N 2.8542(5) 1.61352(19) 1.8758(3) 0.0284(10) Uani 1 1 d . . . . . .
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O1A O 2.9446(5) 1.5887(2) 1.7166(3) 0.0435(10) Uani 1 1 d . . . . . .
O2A O 2.9723(5) 1.70026(18) 1.8903(3) 0.0422(10) Uani 1 1 d . . . . . .
O3A O 2.7970(5) 1.51970(19) 1.8865(3) 0.0427(10) Uani 1 1 d . . . . . .
C1C C 2.8403(9) 1.4418(4) 1.4486(5) 0.065(2) Uani 1 1 d . . . . . .
H1CA H 2.7411 1.4560 1.4591 0.097 Uiso 1 1 calc GR . . . .
H1CB H 2.8937 1.4722 1.4225 0.097 Uiso 1 1 calc GR . . . .
H1CC H 2.9068 1.4287 1.5107 0.097 Uiso 1 1 calc GR . . . .
C2C C 2.8088(7) 1.3943(3) 1.3784(4) 0.0419(15) Uani 1 1 d . . . . . .
C3C C 2.8587(9) 1.3414(4) 1.3999(5) 0.063(2) Uani 1 1 d . . . . . .
H3CA H 2.9183 1.3325 1.4636 0.076 Uiso 1 1 calc R . . . .
H3CB H 2.8345 1.3128 1.3515 0.076 Uiso 1 1 calc R . . . .
C4C C 2.7043(7) 1.4075(2) 1.2816(4) 0.0332(12) Uani 1 1 d . . . . . .
C5C C 2.9236(6) 1.3864(2) 1.2010(4) 0.0277(11) Uani 1 1 d . . . . . .
C6C C 2.9707(7) 1.3337(3) 1.1797(4) 0.0385(13) Uani 1 1 d . . . . . .
H6C H 2.8984 1.3032 1.1679 0.046 Uiso 1 1 calc R . . . .
C7C C 3.1247(7) 1.3253(3) 1.1755(5) 0.0540(18) Uani 1 1 d . . . . . .
H7C H 3.1578 1.2891 1.1595 0.065 Uiso 1 1 calc R . . . .
C8C C 3.2292(7) 1.3692(3) 1.1943(6) 0.057(2) Uani 1 1 d . . . . . .
H8C H 3.3352 1.3630 1.1931 0.069 Uiso 1 1 calc R . . . .
C9C C 3.1815(7) 1.4227(3) 1.2152(6) 0.0534(18) Uani 1 1 d . . . . . .
H9C H 3.2539 1.4531 1.2269 0.064 Uiso 1 1 calc R . . . .
C10C C 3.0251(7) 1.4315(3) 1.2190(5) 0.0383(14) Uani 1 1 d . . . . . .
H10C H 2.9904 1.4677 1.2336 0.046 Uiso 1 1 calc R . . . .
C11C C 2.5524(7) 1.3642(3) 1.0648(4) 0.0354(13) Uani 1 1 d . . . . . .
C12C C 2.4531(7) 1.3909(3) 0.9750(4) 0.0394(14) Uani 1 1 d . . . . . .
H12H H 2.3419 1.3915 0.9776 0.047 Uiso 1 1 calc R . . . .
H12I H 2.4622 1.3700 0.9161 0.047 Uiso 1 1 calc R . . . .
C13C C 2.5167(7) 1.4518(3) 0.9736(4) 0.0370(13) Uani 1 1 d . . . . . .
H13E H 2.5677 1.4556 0.9181 0.044 Uiso 1 1 calc R . . . .
C14C C 2.6394(6) 1.4578(3) 1.0684(4) 0.0327(12) Uani 1 1 d . . . . . .
C15C C 2.3936(8) 1.4967(3) 0.9652(5) 0.0588(19) Uani 1 1 d . . . . . .
H15J H 2.3454 1.4938 1.0206 0.088 Uiso 1 1 calc GR . . . .
H15K H 2.3133 1.4915 0.9044 0.088 Uiso 1 1 calc GR . . . .
H15L H 2.4418 1.5340 0.9653 0.088 Uiso 1 1 calc GR . . . .
N1C N 2.7621(5) 1.3957(2) 1.2023(3) 0.0293(10) Uani 1 1 d . . . . . .
N2C N 2.6604(5) 1.4050(2) 1.1120(3) 0.0288(10) Uani 1 1 d . . . . . .
O1C O 2.5732(5) 1.4260(2) 1.2735(3) 0.0441(11) Uani 1 1 d . . . . . .
O2C O 2.7117(5) 1.49948(18) 1.1029(3) 0.0397(10) Uani 1 1 d . . . . . .
O3C O 2.5492(5) 1.31684(19) 1.0952(3) 0.0522(12) Uani 1 1 d . . . . . .

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C1D 0.063(4) 0.034(3) 0.047(4) 0.009(3) 0.009(3) 0.011(3)
C2D 0.026(2) 0.036(3) 0.031(3) 0.001(2) 0.014(2) 0.001(2)
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C3D 0.037(3) 0.045(3) 0.026(3) 0.004(2) 0.008(2) −0.001(3)
C4D 0.032(3) 0.025(3) 0.029(3) −0.002(2) 0.012(2) 0.001(2)
C5D 0.021(2) 0.036(3) 0.019(2) −0.001(2) 0.0039(18) −0.003(2)
C6D 0.034(3) 0.042(3) 0.026(3) 0.006(2) 0.009(2) 0.002(3)
C7D 0.025(3) 0.070(5) 0.035(3) 0.006(3) 0.011(2) 0.005(3)
C8D 0.030(3) 0.074(5) 0.037(3) 0.000(3) 0.006(2) −0.011(3)
C9D 0.038(3) 0.053(4) 0.038(3) −0.001(3) 0.008(3) −0.015(3)
C10D 0.034(3) 0.040(3) 0.026(3) 0.001(2) 0.009(2) −0.004(3)
C11D 0.041(3) 0.050(4) 0.031(3) −0.003(3) 0.014(3) −0.008(3)
C12D 0.037(3) 0.079(5) 0.027(3) −0.005(3) −0.001(2) −0.010(3)
C13D 0.038(10) 0.104(16) 0.040(10) 0.002(10) 0.005(8) 0.011(10)
C14D 0.025(3) 0.083(6) 0.058(4) −0.004(4) −0.010(3) 0.006(3)
C15D 0.062(10) 0.073(11) 0.050(9) −0.008(8) −0.002(7) −0.015(8)
C16D 0.068(9) 0.065(9) 0.051(8) 0.002(6) −0.013(6) 0.024(7)
C17D 0.020(6) 0.068(8) 0.024(6) −0.002(5) 0.000(4) 0.005(5)
N1D 0.022(2) 0.045(3) 0.023(2) −0.0017(19) 0.0033(17) −0.0045(19)
N2D 0.028(2) 0.046(3) 0.025(2) −0.003(2) 0.0021(18) 0.000(2)
O1D 0.0279(19) 0.042(2) 0.046(2) 0.0022(19) 0.0117(17) −0.0073(17)
O2D 0.037(2) 0.069(3) 0.095(4) −0.031(3) −0.003(3) 0.010(2)
O3D 0.058(3) 0.055(3) 0.046(3) −0.013(2) 0.015(2) −0.013(2)
C1B 0.052(4) 0.036(3) 0.032(3) −0.003(2) 0.010(3) −0.006(3)
C2B 0.038(3) 0.027(3) 0.033(3) 0.000(2) 0.017(2) −0.010(2)
C3B 0.041(3) 0.036(3) 0.030(3) −0.004(2) 0.012(2) −0.005(3)
C4B 0.028(3) 0.035(3) 0.039(3) −0.005(2) 0.010(2) 0.001(2)
C5B 0.029(3) 0.044(3) 0.022(2) 0.001(2) 0.005(2) 0.004(2)
C6B 0.040(3) 0.036(3) 0.034(3) −0.001(2) 0.009(2) −0.003(3)
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C8B 0.027(3) 0.061(4) 0.044(3) −0.003(3) 0.007(2) 0.005(3)
C9B 0.036(3) 0.045(3) 0.032(3) 0.000(3) 0.005(2) 0.010(3)
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C14B 0.035(4) 0.095(7) 0.077(6) −0.042(6) 0.002(4) 0.010(4)
C15B 0.101(10) 0.40(3) 0.088(9) −0.018(15) 0.000(8) −0.033(14)
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O2B 0.037(3) 0.100(5) 0.095(5) −0.040(4) 0.013(3) −0.015(3)
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C7A 0.028(3) 0.058(4) 0.054(4) 0.015(3) 0.007(3) −0.013(3)
C8A 0.030(3) 0.081(5) 0.029(3) 0.005(3) 0.007(2) 0.007(3)
C9A 0.044(4) 0.055(4) 0.051(4) −0.021(3) −0.008(3) 0.016(3)
C10A 0.037(3) 0.040(3) 0.041(3) −0.009(3) 0.001(2) 0.000(3)
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C11A 0.041(3) 0.027(3) 0.043(3) −0.007(3) 0.008(3) 0.004(3)
C12A 0.045(4) 0.046(4) 0.052(4) 0.013(3) −0.001(3) 0.004(3)
C13A 0.027(3) 0.043(3) 0.039(3) −0.006(3) 0.000(2) 0.002(3)
C14A 0.025(3) 0.037(3) 0.036(3) −0.011(3) 0.005(2) 0.001(2)
C15A 0.039(3) 0.067(4) 0.041(3) 0.001(3) 0.004(2) 0.003(3)
N1A 0.023(2) 0.036(2) 0.025(2) −0.0015(19) 0.0046(17) −0.0024(19)
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O2A 0.039(2) 0.035(2) 0.050(2) −0.003(2) 0.0064(19) −0.0097(19)
O3A 0.050(2) 0.034(2) 0.045(2) −0.0036(19) 0.015(2) −0.001(2)
C1C 0.068(5) 0.094(6) 0.030(3) −0.016(4) 0.008(3) −0.011(4)
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C3C 0.075(5) 0.077(6) 0.031(3) 0.018(3) −0.001(3) −0.007(4)
C4C 0.036(3) 0.034(3) 0.032(3) 0.002(2) 0.011(2) −0.008(2)
C5C 0.026(3) 0.032(3) 0.023(2) 0.000(2) 0.003(2) 0.003(2)
C6C 0.028(3) 0.035(3) 0.045(3) −0.004(3) −0.003(2) 0.001(2)
C7C 0.032(3) 0.053(4) 0.071(5) −0.012(4) 0.000(3) 0.014(3)
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C9C 0.031(3) 0.049(4) 0.077(5) −0.008(3) 0.007(3) −0.013(3)
C10C 0.031(3) 0.032(3) 0.050(3) −0.007(3) 0.006(3) −0.005(2)
C11C 0.030(3) 0.036(3) 0.037(3) 0.001(3) 0.003(2) 0.000(2)
C12C 0.033(3) 0.046(4) 0.035(3) 0.000(3) 0.001(2) 0.001(3)
C13C 0.039(3) 0.038(3) 0.033(3) 0.002(3) 0.007(2) 0.002(3)
C14C 0.030(3) 0.033(3) 0.035(3) 0.004(3) 0.007(2) 0.003(3)
C15C 0.060(4) 0.049(4) 0.055(4) 0.003(3) −0.010(3) 0.019(3)
N1C 0.026(2) 0.035(3) 0.025(2) 0.0045(19) 0.0021(17) −0.0019(19)
N2C 0.025(2) 0.036(3) 0.023(2) 0.0031(18) 0.0000(17) 0.0000(19)
O1C 0.036(2) 0.056(3) 0.045(2) −0.003(2) 0.0188(18) 0.002(2)
O2C 0.047(2) 0.034(2) 0.036(2) −0.0004(18) 0.0072(18) 0.000(2)
O3C 0.049(3) 0.040(3) 0.055(3) 0.007(2) −0.011(2) −0.009(2)
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 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix. The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters are
only
 used when they are defined by crystal symmetry. An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
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N2D  C14D C17D 107.9(8) . . ?
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C14D C17D C16D 107.8(11) . . ?
C14D C17D H17D 110.8 . . ?
C16D C17D C12D 114.7(12) . . ?
C16D C17D H17D 110.8 . . ?
C4D  N1D  C5D  128.4(4) . . ?
C4D  N1D  N2D  116.0(4) . . ?
N2D  N1D  C5D  115.6(4) . . ?
C11D N2D  C14D 114.5(5) . . ?
N1D  N2D  C11D 122.4(5) . . ?
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N1D N2D C14D 121.8(5) . . ?
H1BA C1B H1BB 109.5 . . ?
H1BA C1B H1BC 109.5 . . ?
H1BB C1B H1BC 109.5 . . ?
C2B C1B H1BA 109.5 . . ?
C2B C1B H1BB 109.5 . . ?
C2B C1B H1BC 109.5 . . ?
C1B C2B C4B 112.7(5) . . ?
C3B C2B C1B 124.7(5) . . ?
C3B C2B C4B 122.4(5) . . ?
C2B C3B H3BA 120.0 . . ?
C2B C3B H3BB 120.0 . . ?
H3BA C3B H3BB 120.0 . . ?
N1B C4B C2B 116.8(5) . . ?
O1B C4B C2B 122.3(5) . . ?
O1B C4B N1B 120.9(5) . . ?
C6B C5B C10B 121.8(5) . . ?
C6B C5B N1B 120.0(6) . . ?
C10B C5B N1B 118.2(5) . . ?
C5B C6B H6B 120.3 . . ?
C5B C6B C7B 119.3(6) . . ?
C7B C6B H6B 120.3 . . ?
C6B C7B H7B 120.1 . . ?
C8B C7B C6B 119.9(6) . . ?
C8B C7B H7B 120.1 . . ?
C7B C8B H8B 119.6 . . ?
C7B C8B C9B 120.8(6) . . ?
C9B C8B H8B 119.6 . . ?
C8B C9B H9B 119.9 . . ?
C8B C9B C10B 120.3(6) . . ?
C10B C9B H9B 119.9 . . ?
C5B C10B C9B 117.8(6) . . ?
C5B C10B H10B 121.1 . . ?
C9B C10B H10B 121.1 . . ?
N2B C11B C12B 107.9(11) . . ?
O3B C11B C12B 127.0(10) . . ?
O3B C11B N2B 125.0(7) . . ?
C11B C12B H12C 110.9 . . ?
C11B C12B H12E 110.9 . . ?
C11B C12B C13B 104.3(9) . . ?
H12C C12B H12E 108.9 . . ?
C13B C12B H12C 110.9 . . ?
C13B C12B H12E 110.9 . . ?
C12B C13B H13C 108.7 . . ?
C12B C13B C14B 106.6(8) . . ?
C14B C13B H13C 108.7 . . ?
C15B C13B C12B 112.7(15) . . ?
C15B C13B H13C 108.7 . . ?
C15B C13B C14B 111.3(13) . . ?
N2B C14B C13B 104.9(10) . . ?
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O2B C14B C13B 130.3(10) . . ?
O2B C14B N2B 124.8(8) . . ?
C13B C15B H15D 109.5 . . ?
C13B C15B H15E 109.5 . . ?
C13B C15B H15F 109.5 . . ?
H15D C15B H15E 109.5 . . ?
H15D C15B H15F 109.5 . . ?
H15E C15B H15F 109.5 . . ?
C4B N1B C5B 127.6(5) . . ?
C4B N1B N2B 115.5(5) . . ?
N2B N1B C5B 116.5(5) . . ?
C14B N2B C11B 115.1(7) . . ?
C14B N2B N1B 123.2(7) . . ?
N1B N2B C11B 120.6(7) . . ?
H1AA C1A H1AB 109.5 . . ?
H1AA C1A H1AC 109.5 . . ?
H1AB C1A H1AC 109.5 . . ?
C2A C1A H1AA 109.5 . . ?
C2A C1A H1AB 109.5 . . ?
C2A C1A H1AC 109.5 . . ?
C1A C2A C4A 113.1(6) . . ?
C3A C2A C1A 125.1(7) . . ?
C3A C2A C4A 121.5(7) . . ?
C2A C3A H3AA 120.0 . . ?
C2A C3A H3AB 120.0 . . ?
H3AA C3A H3AB 120.0 . . ?
N1A C4A C2A 115.7(5) . . ?
O1A C4A C2A 123.4(5) . . ?
O1A C4A N1A 120.9(5) . . ?
C6A C5A C10A 120.7(5) . . ?
C6A C5A N1A 120.2(5) . . ?
C10A C5A N1A 119.2(5) . . ?
C5A C6A H6A 120.8 . . ?
C5A C6A C7A 118.4(6) . . ?
C7A C6A H6A 120.8 . . ?
C6A C7A H7A 119.4 . . ?
C8A C7A C6A 121.2(6) . . ?
C8A C7A H7A 119.4 . . ?
C7A C8A H8A 119.9 . . ?
C7A C8A C9A 120.2(6) . . ?
C9A C8A H8A 119.9 . . ?
C8A C9A H9A 119.8 . . ?
C8A C9A C10A 120.4(7) . . ?
C10A C9A H9A 119.8 . . ?
C5A C10A H10A 120.5 . . ?
C9A C10A C5A 119.1(6) . . ?
C9A C10A H10A 120.5 . . ?
N2A C11A C12A 107.2(5) . . ?
O3A C11A C12A 129.4(6) . . ?
O3A C11A N2A 123.3(6) . . ?

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C11A C12A H12F 110.9 . . ?
C11A C12A H12G 110.9 . . ?
C11A C12A C13A 104.5(5) . . ?
H12F C12A H12G 108.9 . . ?
C13A C12A H12F 110.9 . . ?
C13A C12A H12G 110.9 . . ?
C12A C13A H13D 109.1 . . ?
C14A C13A C12A 106.5(5) . . ?
C14A C13A H13D 109.1 . . ?
C14A C13A C15A 110.8(5) . . ?
C15A C13A C12A 112.2(5) . . ?
C15A C13A H13D 109.1 . . ?
N2A C14A C13A 106.4(5) . . ?
O2A C14A C13A 128.8(6) . . ?
O2A C14A N2A 124.8(5) . . ?
C13A C15A H15G 109.5 . . ?
C13A C15A H15H 109.5 . . ?
C13A C15A H15I 109.5 . . ?
H15G C15A H15H 109.5 . . ?
H15G C15A H15I 109.5 . . ?
H15H C15A H15I 109.5 . . ?
C4A N1A C5A 125.9(4) . . ?
C4A N1A N2A 116.0(4) . . ?
N2A N1A C5A 116.6(4) . . ?
C14A N2A C11A 115.0(5) . . ?
C14A N2A N1A 121.6(5) . . ?
N1A N2A C11A 122.6(5) . . ?
H1CA C1C H1CB 109.5 . . ?
H1CA C1C H1CC 109.5 . . ?
H1CB C1C H1CC 109.5 . . ?
C2C C1C H1CA 109.5 . . ?
C2C C1C H1CB 109.5 . . ?
C2C C1C H1CC 109.5 . . ?
C1C C2C C4C 115.6(6) . . ?
C3C C2C C1C 124.1(6) . . ?
C3C C2C C4C 120.1(6) . . ?
C2C C3C H3CA 120.0 . . ?
C2C C3C H3CB 120.0 . . ?
H3CA C3C H3CB 120.0 . . ?
N1C C4C C2C 116.3(5) . . ?
O1C C4C C2C 121.6(6) . . ?
O1C C4C N1C 122.1(5) . . ?
C6C C5C C10C 121.9(5) . . ?
C6C C5C N1C 119.5(5) . . ?
C10C C5C N1C 118.6(5) . . ?
C5C C6C H6C 120.3 . . ?
C5C C6C C7C 119.4(6) . . ?
C7C C6C H6C 120.3 . . ?
C6C C7C H7C 120.0 . . ?
C8C C7C C6C 120.0(7) . . ?
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C8C C7C H7C 120.0 . . ?
C7C C8C H8C 119.7 . . ?
C7C C8C C9C 120.6(6) . . ?
C9C C8C H8C 119.7 . . ?
C8C C9C H9C 120.2 . . ?
C8C C9C C10C 119.5(6) . . ?
C10C C9C H9C 120.2 . . ?
C5C C10C C9C 118.5(6) . . ?
C5C C10C H10C 120.8 . . ?
C9C C10C H10C 120.8 . . ?
N2C C11C C12C 107.6(5) . . ?
O3C C11C C12C 129.6(6) . . ?
O3C C11C N2C 122.8(5) . . ?
C11C C12C H12H 110.7 . . ?
C11C C12C H12I 110.7 . . ?
C11C C12C C13C 105.4(5) . . ?
H12H C12C H12I 108.8 . . ?
C13C C12C H12H 110.7 . . ?
C13C C12C H12I 110.7 . . ?
C12C C13C H13E 109.1 . . ?
C14C C13C C12C 105.1(5) . . ?
C14C C13C H13E 109.1 . . ?
C15C C13C C12C 113.8(5) . . ?
C15C C13C H13E 109.1 . . ?
C15C C13C C14C 110.5(5) . . ?
N2C C14C C13C 107.3(5) . . ?
O2C C14C C13C 128.9(5) . . ?
O2C C14C N2C 123.7(5) . . ?
C13C C15C H15J 109.5 . . ?
C13C C15C H15K 109.5 . . ?
C13C C15C H15L 109.5 . . ?
H15J C15C H15K 109.5 . . ?
H15J C15C H15L 109.5 . . ?
H15K C15C H15L 109.5 . . ?
C4C N1C C5C 127.2(4) . . ?
C4C N1C N2C 115.9(4) . . ?
N2C N1C C5C 115.5(4) . . ?
C14C N2C C11C 114.0(4) . . ?
C14C N2C N1C 122.6(5) . . ?
N1C N2C C11C 122.6(5) . . ?

loop_
  _geom_torsion_atom_site_label_1
  _geom_torsion_atom_site_label_2
  _geom_torsion_atom_site_label_3
  _geom_torsion_atom_site_label_4
  _geom_torsion
  _geom_torsion_site_symmetry_1
  _geom_torsion_site_symmetry_2
  _geom_torsion_site_symmetry_3
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_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
C1D C2D C4D N1D 127.6(6) . . . . ?
C1D C2D C4D O1D −52.8(7) . . . . ?
C2D C4D N1D C5D −3.1(8) . . . . ?
C2D C4D N1D N2D 177.9(5) . . . . ?
C3D C2D C4D N1D −56.8(7) . . . . ?
C3D C2D C4D O1D 122.7(6) . . . . ?
C4D N1D N2D C11D 83.5(6) . . . . ?
C4D N1D N2D C14D −82.4(7) . . . . ?
C5D C6D C7D C8D −0.3(9) . . . . ?
C5D N1D N2D C11D −95.6(6) . . . . ?
C5D N1D N2D C14D 98.5(7) . . . . ?
C6D C5D C10D C9D −0.6(8) . . . . ?
C6D C5D N1D C4D −74.5(7) . . . . ?
C6D C5D N1D N2D 104.5(6) . . . . ?
C6D C7D C8D C9D 1.5(10) . . . . ?
C7D C8D C9D C10D −2.2(10) . . . . ?
C8D C9D C10D C5D 1.8(9) . . . . ?
C10D C5D C6D C7D −0.2(8) . . . . ?
C10D C5D N1D C4D 105.5(7) . . . . ?
C10D C5D N1D N2D −75.6(6) . . . . ?
C11D C12D C13D C14D 19.3(16) . . . . ?
C11D C12D C17D C14D −25.8(11) . . . . ?
C11D C12D C17D C16D −141.8(10) . . . . ?
C12D C11D N2D C14D −7.6(7) . . . . ?
C12D C11D N2D N1D −174.5(5) . . . . ?
C12D C13D C14D N2D −22.8(16) . . . . ?
C12D C13D C14D O2D 173.7(10) . . . . ?
C13D C14D N2D C11D 19.1(12) . . . . ?
C13D C14D N2D N1D −174.0(10) . . . . ?
C15D C12D C13D C14D −106.1(13) . . . . ?
C17D C14D N2D C11D −9.8(10) . . . . ?
C17D C14D N2D N1D 157.1(8) . . . . ?
N1D C5D C6D C7D 179.8(5) . . . . ?
N1D C5D C10D C9D 179.5(5) . . . . ?
N2D C11D C12D C13D −9.4(13) . . . . ?
N2D C11D C12D C15D 117.8(10) . . . . ?
N2D C11D C12D C17D 20.8(9) . . . . ?
N2D C14D C17D C12D 21.8(12) . . . . ?
N2D C14D C17D C16D 142.7(10) . . . . ?
O1D C4D N1D C5D 177.3(5) . . . . ?
O1D C4D N1D N2D −1.7(8) . . . . ?
O2D C14D C17D C12D −172.2(9) . . . . ?
O2D C14D C17D C16D −51.2(17) . . . . ?
O2D C14D N2D C11D −176.2(8) . . . . ?
O2D C14D N2D N1D −9.2(12) . . . . ?
O3D C11D C12D C13D 167.5(13) . . . . ?
O3D C11D C12D C15D −65.3(12) . . . . ?
O3D C11D C12D C17D −162.4(9) . . . . ?
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O3D C11D N2D C14D 175.2(7) . . . . . ?
O3D C11D N2D N1D 8.3(9) . . . . . ?
C1B C2B C4B N1B −129.0(6) . . . . ?
C1B C2B C4B O1B 50.3(8) . . . . . ?
C2B C4B N1B C5B 5.2(9) . . . . ?
C2B C4B N1B N2B 176.9(5) . . . . . ?
C3B C2B C4B N1B 55.8(8) . . . . . ?
C3B C2B C4B O1B −124.9(7) . . . . ?
C4B N1B N2B C11B −77.9(8) . . . . ?
C4B N1B N2B C14B 89.2(8) . . . . . ?
C5B C6B C7B C8B −0.7(9) . . . . . ?
C5B N1B N2B C11B 94.8(8) . . . . . ?
C5B N1B N2B C14B −98.2(8) . . . . ?
C6B C5B C10B C9B −0.7(8) . . . . . ?
C6B C5B N1B C4B 75.4(8) . . . . . ?
C6B C5B N1B N2B −96.2(7) . . . . . ?
C6B C7B C8B C9B −0.2(10) . . . . . ?
C7B C8B C9B C10B 0.6(9) . . . . . ?
C8B C9B C10B C5B −0.1(8) . . . . . ?
C10B C5B C6B C7B 1.1(9) . . . . . ?
C10B C5B N1B C4B −104.5(7) . . . . ?
C10B C5B N1B N2B 83.9(7) . . . . ?
C11B C12B C13B C14B −9.4(12) . . . . ?
C11B C12B C13B C15B −131.8(11) . . . . . ?
C12B C11B N2B C14B 2.6(11) . . . . ?
C12B C11B N2B N1B 170.7(7) . . . . ?
C12B C13B C14B N2B 10.9(11) . . . . ?
C12B C13B C14B O2B −171.1(11) . . . . . ?
C13B C14B N2B C11B −8.5(9) . . . . ?
C13B C14B N2B N1B −176.2(6) . . . . ?
C15B C13B C14B N2B 134.2(15) . . . . ?
C15B C13B C14B O2B −47.8(18) . . . . ?
N1B C5B C6B C7B −178.7(5) . . . . ?
N1B C5B C10B C9B 179.1(5) . . . . ?
N2B C11B C12B C13B 4.7(12) . . . . ?
O1B C4B N1B C5B −174.1(6) . . . . ?
O1B C4B N1B N2B −2.5(9) . . . . ?
O2B C14B N2B C11B 173.3(9) . . . . ?
O2B C14B N2B N1B 5.6(13) . . . . ?
O3B C11B C12B C13B −176.9(11) . . . . ?
O3B C11B N2B C14B −175.9(10) . . . . ?
O3B C11B N2B N1B −7.8(13) . . . . ?
C1A C2A C4A N1A −125.1(6) . . . . ?
C1A C2A C4A O1A 57.4(9) . . . . ?
C2A C4A N1A C5A 17.9(8) . . . . ?
C2A C4A N1A N2A −176.9(5) . . . . ?
C3A C2A C4A N1A 61.1(9) . . . . ?
C3A C2A C4A O1A −116.4(8) . . . . ?
C4A N1A N2A C11A −81.7(7) . . . . ?
C4A N1A N2A C14A 87.5(6) . . . . . ?
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C5A C6A C7A C8A −0.3(10) . . . . . ?
C5A N1A N2A C11A 85.0(6) . . . . . ?
C5A N1A N2A C14A −105.8(6) . . . . . ?
C6A C5A C10A C9A 0.7(9) . . . . . ?
C6A C5A N1A C4A 67.6(8) . . . . . ?
C6A C5A N1A N2A −97.5(6) . . . . . ?
C6A C7A C8A C9A 0.6(10) . . . . . ?
C7A C8A C9A C10A −0.2(10) . . . . . ?
C8A C9A C10A C5A −0.5(10) . . . . . ?
C10A C5A C6A C7A −0.3(9) . . . . . ?
C10A C5A N1A C4A −112.7(6) . . . . . ?
C10A C5A N1A N2A 82.2(6) . . . . . ?
C11A C12A C13A C14A 4.6(7) . . . . . ?
C11A C12A C13A C15A −116.8(6) . . . . . ?
C12A C11A N2A C14A 6.9(7) . . . . . ?
C12A C11A N2A N1A 176.7(5) . . . . . ?
C12A C13A C14A N2A −0.8(6) . . . . . ?
C12A C13A C14A O2A −178.8(6) . . . . . ?
C13A C14A N2A C11A −3.8(6) . . . . . ?
C13A C14A N2A N1A −173.8(4) . . . . . ?
C15A C13A C14A N2A 121.4(5) . . . . . ?
C15A C13A C14A O2A −56.6(8) . . . . . ?
N1A C5A C6A C7A 179.4(5) . . . . . ?
N1A C5A C10A C9A −179.0(5) . . . . . ?
N2A C11A C12A C13A −6.7(7) . . . . . ?
O1A C4A N1A C5A −164.5(6) . . . . . ?
O1A C4A N1A N2A 0.7(8) . . . . . ?
O2A C14A N2A C11A 174.3(5) . . . . . ?
O2A C14A N2A N1A 4.3(8) . . . . . ?
O3A C11A C12A C13A 174.2(6) . . . . . ?
O3A C11A N2A C14A −174.0(6) . . . . . ?
O3A C11A N2A N1A −4.1(9) . . . . . ?
C1C C2C C4C N1C 125.3(6) . . . . . ?
C1C C2C C4C O1C −56.9(8) . . . . . ?
C2C C4C N1C C5C −17.9(8) . . . . . ?
C2C C4C N1C N2C 176.4(5) . . . . . ?
C3C C2C C4C N1C −59.5(8) . . . . . ?
C3C C2C C4C O1C 118.3(8) . . . . . ?
C4C N1C N2C C11C −86.3(6) . . . . . ?
C4C N1C N2C C14C 83.1(6) . . . . . ?
C5C C6C C7C C8C 1.2(11) . . . . . ?
C5C N1C N2C C11C 106.3(6) . . . . . ?
C5C N1C N2C C14C −84.3(6) . . . . . ?
C6C C5C C10C C9C −0.1(9) . . . . . ?
C6C C5C N1C C4C 112.1(6) . . . . . ?
C6C C5C N1C N2C −82.1(6) . . . . . ?
C6C C7C C8C C9C −1.7(12) . . . . . ?
C7C C8C C9C C10C 1.3(12) . . . . . ?
C8C C9C C10C C5C −0.4(11) . . . . . ?
C10C C5C C6C C7C −0.3(9) . . . . . ?
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C10C C5C N1C C4C −69.6(7) . . . . . ?
C10C C5C N1C N2C 96.2(6) . . . . . ?
C11C C12C C13C C14C −5.2(6) . . . . . ?
C11C C12C C13C C15C −126.1(6) . . . . . ?
C12C C11C N2C C14C 4.3(7) . . . . . ?
C12C C11C N2C N1C 174.5(5) . . . . . ?
C12C C13C C14C N2C 7.7(6) . . . . . ?
C12C C13C C14C O2C −173.4(6) . . . . . ?
C13C C14C N2C C11C −7.7(6) . . . . . ?
C13C C14C N2C N1C −178.0(5) . . . . . ?
C15C C13C C14C N2C 130.8(6) . . . . . ?
C15C C13C C14C O2C −50.3(8) . . . . . ?
N1C C5C C6C C7C 177.9(5) . . . . . ?
N1C C5C C10C C9C −178.4(6) . . . . . ?
N2C C11C C12C C13C 1.0(6) . . . . . ?
O1C C4C N1C C5C 164.4(6) . . . . . ?
O1C C4C N1C N2C −1.3(8) . . . . . ?
O2C C14C N2C C11C 173.3(5) . . . . . ?
O2C C14C N2C N1C 3.1(9) . . . . . ?
O3C C11C C12C C13C −178.1(7) . . . . . ?
O3C C11C N2C C14C −176.6(6) . . . . . ?
O3C C11C N2C N1C −6.3(9) . . . . . ?

_shelx_res_file
;
    cu_axi20_0m.res created by SHELXL−2014/7

TITL cu_axi20_0m in P2(1)
CELL 1.54178 8.8088 23.665 14.1477 90 104.096 90
ZERR 8 0.0004 0.0011 0.0007 0 0.002 0
LATT −1
SYMM −X,0.5+Y,−Z
SFAC C H N O
UNIT 120 128 16 24

L.S. 7
PLAN  −5 0 0
SIZE 0.148 0.174 0.24
TEMP −173.14
HTAB
BOND $H
CONF
fmap 2
acta
REM <olex2.extras>
REM <HklSrc "%.\\cu_axi20_0m.hkl">
REM </olex2.extras>

WGHT    0.073000    1.915400
FVAR       0.49180    0.54849
```

```
C1D    1     2.736052     1.749920     1.075647     11.00000     0.06265
0.03416 =
          0.04661     0.00919     0.00855     0.01095
AFIX 137
H1DA   2     2.657682     1.751940     1.013295     11.00000    -1.50000
H1DB   2     2.838546     1.760882     1.066068     11.00000    -1.50000
H1DC   2     2.706589     1.775663     1.122439     11.00000    -1.50000
AFIX   0
C2D    1     2.744245     1.690486     1.114232     11.00000     0.02607
0.03575 =
          0.03053     0.00112     0.01374     0.00061
C3D    1     2.665895     1.646887     1.064645     11.00000     0.03661
0.04538 =
          0.02621     0.00420     0.00773    -0.00081
AFIX  93
H3DA   2     2.681879     1.609758     1.090704     11.00000    -1.20000
H3DB   2     2.594390     1.653119     1.003447     11.00000    -1.20000
AFIX   0
C4D    1     2.856610     1.681908     1.209981     11.00000     0.03214
0.02511 =
          0.02867    -0.00178     0.01186     0.00096
C5D    1     2.644240     1.638767     1.279943     11.00000     0.02145
0.03563 =
          0.01923    -0.00073     0.00391    -0.00288
C6D    1     2.531756     1.678948     1.285051     11.00000     0.03388
0.04188 =
          0.02578     0.00580     0.00933     0.00233
AFIX  43
H6D    2     2.556826     1.718048     1.290360     11.00000    -1.20000
AFIX   0
C7D    1     2.381642     1.660172     1.282154     11.00000     0.02503
0.06984 =
          0.03521     0.00553     0.01061     0.00543
AFIX  43
H7D    2     2.302505     1.686777     1.285797     11.00000    -1.20000
AFIX   0
C8D    1     2.345540     1.603252     1.274036     11.00000     0.02952
0.07383 =
          0.03651     0.00033     0.00601    -0.01109
AFIX  43
H8D    2     2.241613     1.591028     1.270607     11.00000    -1.20000
AFIX   0
C9D    1     2.460031     1.564377     1.270929     11.00000     0.03760
0.05258 =
          0.03793    -0.00119     0.00773    -0.01538
AFIX  43
H9D    2     2.435901     1.525191     1.267681     11.00000    -1.20000
AFIX   0
C10D   1     2.610270     1.582048     1.272509     11.00000     0.03438
0.04002 =
```

```
        0.02603    0.00084    0.00921   -0.00371
AFIX  43
H10D  2    2.688930    1.555319    1.268508    11.00000   -1.20000
AFIX   0
C11D  1    2.953502    1.689495    1.441393    11.00000    0.04107
0.04997 =
        0.03099   -0.00330    0.01385   -0.00761
C12D  1    3.086494    1.663494    1.518686    11.00000    0.03713
0.07864 =
        0.02712   -0.00469   -0.00094   -0.00969
PART 2
AFIX  13
H12D  2    3.056554    1.666285    1.582317   -21.00000   -1.20000
AFIX  23
PART 0
PART 1
H12A  2    3.047339    1.648307    1.573512    21.00000   -1.20000
H12B  2    3.168801    1.691876    1.544297    21.00000   -1.20000
AFIX   0
PART 0
PART 2
C13D  1    3.103107    1.604677    1.499745   -21.00000    0.03832
0.10398 =
        0.03960    0.00165    0.00538    0.01094
AFIX  23
H13A  2    3.052428    1.580718    1.540578   -21.00000   -1.20000
H13B  2    3.214555    1.593793    1.510667   -21.00000   -1.20000
AFIX   0
PART 0
C14D  1    3.012077    1.600519    1.384392    11.00000    0.02522
0.08347 =
        0.05777   -0.00443   -0.01047    0.00627
PART 2
C15D  1    3.227923    1.695494    1.528168   -21.00000    0.06194
0.07348 =
        0.05005   -0.00817   -0.00174   -0.01468
AFIX 137
H15A  2    3.253005    1.698210    1.464531   -21.00000   -1.50000
H15B  2    3.313876    1.676600    1.574446   -21.00000   -1.50000
H15C  2    3.213407    1.733510    1.551998   -21.00000   -1.50000
AFIX   0
PART 0
PART 1
C16D  1    3.199507    1.561833    1.523906    21.00000    0.06842
0.06513 =
        0.05070    0.00235   -0.01336    0.02390
AFIX 137
H16A  2    3.290185    1.570100    1.577762    21.00000   -1.50000
H16B  2    3.226983    1.532194    1.482630    21.00000   -1.50000
H16C  2    3.112274    1.548946    1.550157    21.00000   -1.50000
```

```
AFIX    0
C17D  1    3.151936    1.614536    1.464171    21.00000    0.02014
0.06817 =
          0.02374   -0.00189   -0.00029    0.00455
AFIX  13
H17D  2    3.239605    1.628508    1.436882    21.00000   -1.20000
AFIX    0
PART 0
N1D   3    2.801946    1.656557    1.282342    11.00000    0.02240
0.04508 =
          0.02349   -0.00170    0.00326   -0.00453
N2D   3    2.911913    1.647451    1.370140    11.00000    0.02823
0.04581 =
          0.02507   -0.00333    0.00207   -0.00042
O1D   4    2.994057    1.696570    1.224232    11.00000    0.02794
0.04158 =
          0.04615    0.00216    0.01170   -0.00727
O2D   4    3.000270    1.561439    1.328655    11.00000    0.03716
0.06884 =
          0.09520   -0.03113   -0.00332    0.01021
O3D   4    2.887519    1.733552    1.437541    11.00000    0.05819
0.05547 =
          0.04558   -0.01291    0.01541   -0.01341

C1B   1    3.227468    1.774883    1.084309    11.00000    0.05211
0.03558 =
          0.03207   -0.00313    0.00954   -0.00614
AFIX 137
H1BA  2    3.161733    1.774197    1.017586    11.00000   -1.50000
H1BB  2    3.184436    1.748882    1.125020    11.00000   -1.50000
H1BC  2    3.334079    1.763188    1.084022    11.00000   -1.50000
AFIX    0
C2B   1    3.230760    1.833461    1.124444    11.00000    0.03771
0.02652 =
          0.03292   -0.00027    0.01653   -0.00954
C3B   1    3.151831    1.876812    1.077675    11.00000    0.04070
0.03634 =
          0.02994   -0.00355    0.01217   -0.00533
AFIX  93
H3BA  2    3.087250    1.871864    1.013849    11.00000   -1.20000
H3BB  2    3.159724    1.912896    1.107910    11.00000   -1.20000
AFIX    0
C4B   1    3.345031    1.839729    1.221608    11.00000    0.02755
0.03485 =
          0.03859   -0.00512    0.00954    0.00066
C5B   1    3.130246    1.879851    1.292239    11.00000    0.02917
0.04375 =
          0.02199    0.00099    0.00510    0.00374
C6B   1    3.020684    1.838917    1.297339    11.00000    0.04003
0.03647 =
```

```
          0.03433   −0.00096    0.00924   −0.00286
AFIX  43
H6B   2    3.049975     1.800216     1.304062     11.00000    −1.20000
AFIX   0
C7B   1    2.867574     1.854889     1.292547     11.00000     0.04029
0.05264 =
          0.04555   −0.01230    0.01697   −0.01586
AFIX  43
H7B   2    2.791012     1.827071     1.295298     11.00000    −1.20000
AFIX   0
C8B   1    2.826677     1.911164     1.283792     11.00000     0.02735
0.06082 =
          0.04369   −0.00334    0.00712    0.00471
AFIX  43
H8B   2    2.721551     1.921909     1.280329     11.00000    −1.20000
AFIX   0
C9B   1    2.936331     1.952065     1.279993     11.00000     0.03567
0.04454 =
          0.03211    0.00035    0.00520    0.01020
AFIX  43
H9B   2    2.906581     1.990741     1.274569     11.00000    −1.20000
AFIX   0
C10B  1    3.092141     1.936809     1.284110     11.00000     0.03191
0.03250 =
          0.03005    0.00133    0.00602    0.00077
AFIX  43
H10B  2    3.168773     1.964609     1.281411     11.00000    −1.20000
AFIX   0
C11B  1    3.428730     1.818291     1.444655     11.00000     0.07708
0.10968 =
          0.04551   −0.00819   −0.01635    0.06504
C12B  1    3.564577     1.831768     1.527864     11.00000     0.10681
0.13421 =
          0.08206   −0.00917   −0.04464    0.05797
AFIX  23
H12C  2    3.656710     1.808460     1.525069     11.00000    −1.20000
H12E  2    3.537932     1.825118     1.591009     11.00000    −1.20000
AFIX   0
C13B  1    3.596449     1.893673     1.514771     11.00000     0.04050
0.20049 =
          0.07305   −0.06076   −0.02857    0.02397
AFIX  13
H13C  2    3.562117     1.915810     1.566091     11.00000    −1.20000
AFIX   0
C14B  1    3.494674     1.910926     1.414224     11.00000     0.03534
0.09536 =
          0.07733   −0.04225    0.00182    0.00974
C15B  1    3.752294     1.904631     1.521895     11.00000     0.10056
0.40120 =
          0.08807   −0.01801   −0.00016   −0.03319
```

```
AFIX 137
H15D   2    3.765980    1.944854    1.509716    11.00000   -1.50000
H15E   2    3.788408    1.882008    1.473607    11.00000   -1.50000
H15F   2    3.813450    1.894833    1.587433    11.00000   -1.50000
AFIX   0
N1B    3    3.289421    1.863757    1.295111    11.00000    0.02921
0.05047 =
           0.03102   -0.01007    0.00015    0.00938
N2B    3    3.395620    1.866421    1.385453    11.00000    0.03732
0.06596 =
           0.03946   -0.01721   -0.00942    0.01585
O1B    4    3.480117    1.824063    1.236003    11.00000    0.03047
0.04814 =
           0.05357   -0.01289    0.01014    0.00494
O2B    4    3.493578    1.952853    1.369672    11.00000    0.03699
0.09952 =
           0.09464   -0.03978    0.01292   -0.01537
O3B    4    3.359614    1.773472    1.428483    11.00000    0.15270
0.07344 =
           0.06390    0.00495   -0.00725    0.05863


C1A    1    2.677077    1.571391    1.540316    11.00000    0.08441
0.09612 =
           0.04059   -0.01974    0.01432   -0.02623
AFIX 137
H1AA   2    2.624730    1.583584    1.474214    11.00000   -1.50000
H1AB   2    2.609586    1.544807    1.564138    11.00000   -1.50000
H1AC   2    2.776161    1.552822    1.539428    11.00000   -1.50000
AFIX   0
C2A    1    2.708821    1.621810    1.606348    11.00000    0.03940
0.07283 =
           0.02530    0.00184    0.01617   -0.00892
C3A    1    2.665417    1.673475    1.580728    11.00000    0.08108
0.08183 =
           0.04455    0.02451    0.01964   -0.00012
AFIX  93
H3AA   2    2.609131    1.681319    1.515789    11.00000   -1.20000
H3AB   2    2.690123    1.703220    1.627028    11.00000   -1.20000
AFIX   0
C4A    1    2.813324    1.608733    1.704843    11.00000    0.03448
0.04050 =
           0.03248   -0.00412    0.01110   -0.00588
C5A    1    2.592546    1.634033    1.781052    11.00000    0.02442
0.03591 =
           0.01995   -0.00010    0.00419   -0.00371
C6A    1    2.484383    1.591204    1.760553    11.00000    0.03730
0.03997 =
           0.03827    0.00081    0.00735   -0.00572
AFIX  43
H6A    2    2.514541    1.554047    1.747400    11.00000   -1.20000
```

```
AFIX    0
C7A    1     2.329602     1.603513     1.759503     11.00000     0.02817
0.05754 =
         0.05444     0.01457     0.00652    -0.01276
AFIX   43
H7A    2     2.253278     1.574369     1.745080     11.00000    -1.20000
AFIX    0
C8A    1     2.285810     1.656169     1.778623     11.00000     0.03001
0.08148 =
         0.02852     0.00459     0.00682     0.00729
AFIX   43
H8A    2     2.179658     1.663640     1.778131     11.00000    -1.20000
AFIX    0
C9A    1     2.393618     1.698631     1.798630     11.00000     0.04447
0.05470 =
         0.05135    -0.02095    -0.00833     0.01595
AFIX   43
H9A    2     2.362062     1.735589     1.811846     11.00000    -1.20000
AFIX    0
C10A   1     2.548164     1.688254     1.799798     11.00000     0.03725
0.03975 =
         0.04093    -0.00890     0.00068     0.00046
AFIX   43
H10A   2     2.623137     1.717874     1.813269     11.00000    -1.20000
AFIX    0
C11A   1     2.871711     1.560383     1.920628     11.00000     0.04104
0.02742 =
         0.04277    -0.00683     0.00802     0.00442
C12A   1     2.995062     1.566168     2.015344     11.00000     0.04533
0.04640 =
         0.05213     0.01341    -0.00052     0.00405
AFIX   23
H12F   2     3.079154     1.537860     2.019388     11.00000    -1.20000
H12G   2     2.948612     1.561224     2.071872     11.00000    -1.20000
AFIX    0
C13A   1     3.059119     1.626639     2.012557     11.00000     0.02690
0.04277 =
         0.03885    -0.00635     0.00048     0.00212
AFIX   13
H13D   2     3.044924     1.647971     2.070814     11.00000    -1.20000
AFIX    0
C14A   1     2.961230     1.654029     1.921307     11.00000     0.02509
0.03712 =
         0.03632    -0.01090     0.00476     0.00135
C15A   1     3.231537     1.627046     2.011890     11.00000     0.03871
0.06675 =
         0.04073     0.00090     0.00391     0.00281
AFIX  137
H15G   2     3.247792     1.602966     1.958861     11.00000    -1.50000
H15H   2     3.293858     1.612661     2.074283     11.00000    -1.50000
```

```
H15I   2      3.264041      1.665772      2.002096      11.00000     −1.50000
AFIX    0
N1A    3      2.754398      1.622929      1.783789      11.00000      0.02332
0.03574 =
         0.02499     −0.00152      0.00464     −0.00239
N2A    3      2.854217      1.613518      1.875783      11.00000      0.02551
0.02798 =
         0.03049     −0.00220      0.00428      0.00058
O1A    4      2.944570      1.588682      1.716560      11.00000      0.03251
0.05521 =
         0.04731     −0.00439      0.01868      0.00160
O2A    4      2.972265      1.700258      1.890345      11.00000      0.03897
0.03472 =
         0.05047     −0.00338      0.00644     −0.00970
O3A    4      2.796976      1.519701      1.886504      11.00000      0.05050
0.03436 =
         0.04495     −0.00360      0.01493     −0.00146


C1C    1      2.840262      1.441798      1.448619      11.00000      0.06827
0.09443 =
         0.02966     −0.01622      0.00820     −0.01118
AFIX 137
H1CA   2      2.741105      1.455972      1.459089      11.00000     −1.50000
H1CB   2      2.893718      1.472203      1.422526      11.00000     −1.50000
H1CC   2      2.906756      1.428660      1.510692      11.00000     −1.50000
AFIX    0
C2C    1      2.808846      1.394293      1.378439      11.00000      0.04355
0.05457 =
         0.02962      0.00365      0.01275     −0.01194
C3C    1      2.858708      1.341441      1.399893      11.00000      0.07482
0.07663 =
         0.03134      0.01826     −0.00148     −0.00663
AFIX  93
H3CA   2      2.918344      1.332471      1.463589      11.00000     −1.20000
H3CB   2      2.834533      1.312824      1.351455      11.00000     −1.20000
AFIX    0
C4C    1      2.704333      1.407475      1.281591      11.00000      0.03558
0.03408 =
         0.03150      0.00249      0.01107     −0.00847
C5C    1      2.923621      1.386390      1.201044      11.00000      0.02627
0.03225 =
         0.02308     −0.00005      0.00331      0.00289
C6C    1      2.970659      1.333655      1.179683      11.00000      0.02841
0.03520 =
         0.04533     −0.00386     −0.00342      0.00148
AFIX  43
H6C    2      2.898387      1.303151      1.167884      11.00000     −1.20000
AFIX    0
C7C    1      3.124657      1.325278      1.175470      11.00000      0.03196
0.05284 =
```

```
         0.07081    -0.01206     0.00026     0.01386
AFIX  43
H7C    2     3.157795     1.289072     1.159501     11.00000    -1.20000
AFIX   0
C8C    1     3.229176     1.369229     1.194341     11.00000     0.02509
0.07066 =
         0.07287    -0.01978     0.00669     0.00361
AFIX  43
H8C    2     3.335217     1.363011     1.193098     11.00000    -1.20000
AFIX   0
C9C    1     3.181478     1.422673     1.215226     11.00000     0.03095
0.04856 =
         0.07725    -0.00825     0.00680    -0.01324
AFIX  43
H9C    2     3.253904     1.453131     1.226920     11.00000    -1.20000
AFIX   0
C10C   1     3.025131     1.431459     1.218969     11.00000     0.03094
0.03192 =
         0.05012    -0.00658     0.00608    -0.00535
AFIX  43
H10C   2     2.990444     1.467708     1.233552     11.00000    -1.20000
AFIX   0
C11C   1     2.552417     1.364250     1.064760     11.00000     0.03036
0.03621 =
         0.03708     0.00079     0.00326     0.00038
C12C   1     2.453135     1.390944     0.974979     11.00000     0.03258
0.04647 =
         0.03528    -0.00048     0.00067     0.00084
AFIX  23
H12H   2     2.341873     1.391495     0.977636     11.00000    -1.20000
H12I   2     2.462172     1.369956     0.916063     11.00000    -1.20000
AFIX   0
C13C   1     2.516674     1.451781     0.973603     11.00000     0.03914
0.03832 =
         0.03278     0.00211     0.00719     0.00213
AFIX  13
H13E   2     2.567696     1.455623     0.918051     11.00000    -1.20000
AFIX   0
C14C   1     2.639410     1.457839     1.068430     11.00000     0.02970
0.03303 =
         0.03511     0.00437     0.00742     0.00328
C15C   1     2.393599     1.496666     0.965246     11.00000     0.06013
0.04918 =
         0.05478     0.00306    -0.00979     0.01854
AFIX 137
H15J   2     2.345413     1.493817     1.020619     11.00000    -1.50000
H15K   2     2.313290     1.491477     0.904376     11.00000    -1.50000
H15L   2     2.441778     1.533996     0.965290     11.00000    -1.50000
AFIX   0
N1C    3     2.762110     1.395656     1.202258     11.00000     0.02601
```

```
0.03490 =
         0.02482    0.00447    0.00206   -0.00191
N2C   3    2.660362    1.405016    1.111961    11.00000    0.02507
0.03557 =
         0.02280    0.00308    0.00004    0.00005
O1C   4    2.573228    1.426003    1.273503    11.00000    0.03605
0.05557 =
         0.04525   -0.00270    0.01884    0.00170
O2C   4    2.711662    1.499485    1.102948    11.00000    0.04728
0.03399 =
         0.03632   -0.00040    0.00718   -0.00039
O3C   4    2.549154    1.316842    1.095215    11.00000    0.04908
0.04031 =
         0.05516    0.00698   -0.01085   -0.00945
HKLF 4

REM  cu_axi20_0m in P2(1)
REM R1 =  0.0610 for    7549 Fo > 4sig(Fo)  and  0.0700 for all
8826 data
REM    749 parameters refined using      1 restraints

END

WGHT      0.0730       1.9153

REM Instructions for potential hydrogen bonds
EQIV $1 x, y, z+1
HTAB C13A O1D_$1
HTAB C10C O2D
EQIV $2 −x+6, y−1/2, −z+2
HTAB C12C O1B_$2
EQIV $3 x, y, z−1
HTAB C13C O3A_$3

REM Highest difference peak  0.503,  deepest hole −0.294,  1−sigma
level  0.052
Q1    1    3.5740  1.8121  1.6046  11.00000  0.05    0.50
Q2    1    3.0554  1.5395  2.0709  11.00000  0.05    0.43
Q3    1    3.0624  1.5727  1.9708  11.00000  0.05    0.36
Q4    1    3.0399  1.7238  1.6067  11.00000  0.05    0.30
Q5    1    3.0272  1.6994  1.5181  11.00000  0.05    0.25
;
_shelx_res_checksum                   38044
_shelx_hkl_file
;
  −1    0    0 8.50823 0.99998    5
  −1    0    0 9.64630 0.93628    4
  −1    0    0 9.61772 1.05123    5
  −1    0    0 9.65104 0.86998    4
  −2    0    0 372.460 22.1450    4
```

−2	0	0	350.891	22.0149	5
−2	0	0	361.065	22.1294	4
−2	0	0	366.343	22.8874	2
−2	0	0	366.286	22.0786	3
−2	0	0	344.766	21.9958	5
−3	0	0	13.9800	1.85809	4
−3	0	0	13.5872	1.61828	3
−3	0	0	13.8500	1.76228	4
−3	0	0	11.3315	2.48837	2
−4	0	0	172.308	10.7200	3
−4	0	0	166.411	10.7420	4
−4	0	0	156.745	11.2278	1
−4	0	0	159.866	10.7052	4
−5	0	0	31.3220	2.96691	3
−5	0	0	27.8813	3.02505	4
−6	0	0	87.1173	6.55138	4
−8	0	0	33.1615	3.13468	8
9	0	0	1.31977	3.23207	13
−9	0	0	4.71977	1.33444	8
10	0	0	6.08522	2.78165	13
−1	−1	0	3400.10	214.337	5
−1	−1	0	3502.60	212.789	4
−1	−1	0	3554.59	215.064	5
−1	1	0	3512.91	212.636	3
−1	1	0	3373.92	212.638	3
−1	1	0	3766.77	212.880	4
−1	1	0	3488.97	212.824	4
−2	−1	0	592.201	35.5971	3
−2	−1	0	574.487	35.6510	4
−2	−1	0	589.853	36.3377	2
−2	−1	0	586.380	35.6447	4
−2	−1	0	566.510	35.9582	5
−2	−1	0	606.282	36.1380	5
−2	1	0	599.904	35.5110	4
−2	1	0	573.239	36.2869	2
−2	1	0	569.534	35.5087	4
−2	1	0	572.013	35.4463	3
−3	−1	0	101.871	7.25326	5
−3	−1	0	103.758	8.28152	2
−3	−1	0	102.291	7.26293	4
−3	−1	0	99.5698	7.22996	5
−3	−1	0	103.402	7.26932	4
−3	−1	0	104.632	7.19918	3
−3	1	0	124.904	7.17364	4
−3	1	0	119.963	7.16134	4
−3	1	0	119.866	7.09344	3
−4	−1	0	16.1654	2.34681	4
−4	−1	0	15.6828	3.22674	2
−4	−1	0	15.5517	3.17043	1
−5	−1	0	1.20257	1.71842	1

−5	−1	0	0.57397	1.37805	4
−5	1	0	3.71584	1.29843	3
−5	1	0	2.80866	1.37912	4
−6	−1	0	18.2338	2.91189	4
−6	1	0	22.0778	2.56196	4
−8	−1	0	16.0721	2.44913	8
−8	1	0	23.5257	2.66766	8
8	1	0	18.8342	3.87513	13
−9	−1	0	3.65412	1.50227	8
9	1	0	−2.8929	3.24855	13
10	−1	0	3.47242	2.13337	13
10	1	0	1.76557	2.28696	13
0	2	0	13.4162	1.09505	5
0	2	0	13.2145	0.98290	4
−1	−2	0	1454.31	86.9994	5
−1	2	0	1405.82	86.3582	4
−1	2	0	1422.08	86.4258	4
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−2	−2	0	3.39989	1.48861	5
−2	−2	0	3.67705	0.91506	4
−2	−2	0	3.83472	1.01498	4
−2	−2	0	3.26013	0.92040	4
−2	−2	0	3.75505	1.51147	5
−2	−2	0	4.52889	1.83945	2
−3	−2	0	2246.21	138.432	3
−3	−2	0	2136.74	138.850	5
−3	−2	0	2356.36	139.106	5
−3	−2	0	2342.51	139.541	2
−3	−2	0	2328.64	138.534	4
−3	2	0	2278.95	138.171	4
−3	2	0	2359.21	138.174	3
−3	2	0	2201.76	138.181	4
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−4	2	0	23.8026	2.39374	4
−4	2	0	23.4686	2.21688	3
−4	2	0	23.6391	2.35308	4
−5	−2	0	30.2180	3.40611	4
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−5	2	0	26.8185	2.66588	4
−5	2	0	26.8441	2.62063	4
−5	2	0	30.0472	2.84903	3
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−6	2	0	52.7285	4.14124	4
−6	2	0	56.3632	4.87015	3
−7	−2	0	29.7196	3.93449	10
7	2	0	28.8668	5.19295	13

−8	−2	0	10.6367	2.05917	8
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−3	3	0	735.658	43.1091	3
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−2	5	0	251.086	14.8064	3
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−4	−5	0	236.978	17.0512	1
−4	5	0	221.975	14.7948	3
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−6	−5	0	4.67573	2.94242	1
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−6	5	0	1.54434	0.70348	9
−6	5	0	2.24375	1.10782	3
−7	−5	0	214.022	13.2628	10
−7	5	0	168.453	11.6426	9
−7	5	0	170.609	12.4209	7
−7	5	0	178.621	11.5075	6
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−8	−5	0	68.5184	5.57553	8
−8	−5	0	70.3486	6.09436	10
−8	5	0	58.3886	4.26091	6
8	5	0	71.7575	8.91834	13
−8	5	0	66.1142	5.50200	7
−8	5	0	67.5291	4.49833	9
−9	−5	0	50.3308	4.11286	8
9	5	0	58.6869	6.66293	13
−9	5	0	42.8403	3.24730	9
−9	5	0	45.1127	3.11093	6
0	−6	0	16.4135	2.54108	2
0	6	0	16.2073	1.68323	3
0	6	0	14.7093	1.74436	4
0	6	0	14.5199	1.72817	4
−1	−6	0	407.913	26.1522	5
−1	−6	0	401.981	25.8684	2
1	−6	0	405.779	25.7486	2
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1	6	0	393.492	25.9007	5
−1	6	0	419.808	24.8930	4
−1	6	0	414.497	24.8874	4
−1	6	0	410.655	24.9255	3
1	6	0	419.784	25.2759	4
−2	−6	0	94.0629	7.54272	2
−2	−6	0	85.1142	8.01434	5
−2	6	0	109.598	6.05951	3
−3	−6	0	15.7598	3.36335	1
−3	−6	0	16.9501	3.30917	2
−3	−6	0	15.2803	3.49577	5

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−4   −6    0 55.3450 6.57253    5
−4   −6    0 57.2099 6.19644    2
−4   −6    0 59.7286 6.43130    1
−5   −6    0 99.0487 9.14341    1
−5   −6    0 98.3091 8.26162    5
−5   −6    0 94.8660 9.24151    2
−5    6    0 96.8700 6.56228    3
−6   −6    0 34.3785 5.46219    1
−6   −6    0 38.6829 6.54445    2
−6    6    0 34.5223 3.55995    7
−6    6    0 33.5182 3.07223    3
−6    6    0 33.5076 2.34736    6
−6    6    0 35.1201 2.55154    9
−7   −6    0 134.321 10.7599   10
−7    6    0 152.555 10.2900    7
 7    6    0 133.254 11.7623   13
−7    6    0 152.065 9.31806    9
−7    6    0 160.940 9.18720    6
−8    6    0 14.6750 2.41817    7
 8    6    0 −0.5473 3.67989   13
−9   −6    0 18.6199 2.77213    8
 9    6    0 17.4426 4.01508   13
−9    6    0 23.7433 1.89441    9
−9    6    0 22.6976 1.59828    6
 0   −7    0 2.10173 1.81062    2
 0    7    0 3.87742 0.95078    4
 0    7    0 3.93302 0.89370    3
 0    7    0 2.26981 0.87855    4
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−1   −7    0 130.449 9.47604    2
−1    7    0 137.087 8.26158    4
−1    7    0 135.517 8.26167    4
 1    7    0 125.548 8.65432    4
−1    7    0 138.587 8.33143    3
 1    7    0 122.197 8.57740    3
−2   −7    0 88.8690 8.45770    5
−2   −7    0 86.4925 7.74259    2
−2    7    0 113.673 6.38897    3
−2    7    0 110.382 6.42017    3
−3   −7    0 269.204 19.4250    1
−3   −7    0 272.528 19.6454    5
−3   −7    0 279.411 19.1334    2
−3    7    0 294.011 17.1147    3
−4   −7    0 202.162 16.0860    5
−4   −7    0 203.341 15.7870    1
−4   −7    0 205.855 15.6342    2
−4    7    0 233.430 13.2393    3
−5   −7    0 26.3789 5.02637    1
−5   −7    0 32.5367 5.34080    2
−5   −7    0 26.3863 4.39952    5
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−5	7	0	22.4749	1.72436	6
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−6	−7	0	9.23943	3.83740	2
−6	−7	0	8.38194	4.02947	1
−6	7	0	3.07208	0.61647	6
−6	7	0	3.48866	0.71377	9
−6	7	0	4.17389	1.62917	7
−7	−7	0	73.0813	7.90974	1
−7	−7	0	70.3613	6.86430	10
−7	7	0	81.4410	4.91561	6
−7	7	0	82.4345	5.11592	9
−7	7	0	78.8852	6.26966	7
7	7	0	72.8714	8.30510	13
−8	−7	0	2.75698	1.91652	8
−8	−7	0	6.05482	2.32822	10
8	7	0	7.70060	4.23345	13
−8	7	0	3.75479	0.88177	9
−8	7	0	4.61216	0.67329	6
−8	7	0	4.27216	1.87850	7
−9	−7	0	1.93143	1.54806	8
9	7	0	3.32708	3.26443	13
−9	7	0	0.92963	0.63122	9
−9	7	0	0.39507	0.42246	6
0	−8	0	546.511	35.6335	2
0	8	0	571.406	34.8485	3
0	8	0	577.754	34.8634	4
0	8	0	569.187	34.8514	4
0	8	0	569.201	34.8574	3
1	−8	0	47.4853	4.58566	2
−1	−8	0	60.5965	4.97226	2
−1	8	0	49.6716	3.49994	3
1	8	0	57.3204	3.93986	3
−1	8	0	46.3382	3.48235	4
−1	8	0	48.8284	3.54387	3
−1	8	0	46.8005	3.49025	4
2	−8	0	5031.86	309.161	2
−2	−8	0	5297.46	309.417	2
−2	8	0	4998.55	308.390	3
−2	8	0	5037.45	308.302	3
−3	−8	0	29.3873	4.68511	2
−3	8	0	44.8125	2.89374	3
−3	8	0	44.7267	2.83575	3
−4	−8	0	386.100	26.2275	5
−4	−8	0	386.024	25.8033	1
−4	−8	0	392.699	25.5808	2
−4	8	0	360.367	22.7267	3
−4	8	0	337.253	22.7625	3
−5	−8	0	50.8861	7.24802	1
−5	−8	0	45.8443	6.47916	2

−5	8	0	47.9338	3.48459	3
−5	8	0	45.1585	2.96890	6
−5	8	0	46.1733	3.39078	3
−5	8	0	47.8207	2.98089	6
−6	−8	0	154.184	11.8526	10
−6	−8	0	168.271	14.5205	2
−6	−8	0	168.545	14.2561	1
−6	8	0	157.240	11.0694	7
−6	8	0	168.557	9.98978	6
−6	8	0	160.355	10.1042	9
−7	−8	0	17.2110	4.57193	1
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