

Supplementary Materials

Circularly Recyclable Polymers Featuring Topochemically Weakened Carbon-Carbon Bonds

Xuyi Luo^{a§}, Zitang Wei^{a§}, Bumjoon Seo^{a§}, Qixuan Hu^a, Xiaokang Wang^b, Joseph A. Romo^c, Mayank Jain^d, Mukerrem Cakmak^d, Bryan W. Boudouris^{a,c}, Kejie Zhao^b, Jianguo Mei^c, Brett M. Savoie^a, Letian Dou^{a*}

^a*Davidson School of Chemical Engineering, Purdue University, 480 Stadium Mall Drive, West Lafayette, IN 47907, United States*

^b*School of Mechanical Engineering, Purdue University, 585 Purdue Mall, West Lafayette, IN 47907, United States*

^c*Department of Chemistry, Purdue University, 560 Oval Drive, West Lafayette, IN 47907, United States*

^d*School of Materials Engineering, Purdue University, 701 W Stadium Ave, West Lafayette, IN 47907, United States*

*Email: dou10@purdue.edu

§X.L., Z.W. and B.S. contributed equally to this paper.

This file includes:

- I. Methods
- II. Supplementary Figures and Tables
- III. Appendix (single crystal structure data)

I. Methods

Materials and Characterizations

All reagents were purchased from suppliers and used without further purification. ^1H - and ^{13}C -NMR spectra were recorded using a Bruker ARX 400 spectrometer where the samples were dissolved in deuterated chloroform at 298 K. Polymer molecular weights were estimated by size exclusion chromatography (SEC) using a TOSOH Bioscience EcoSEC SEC system. The size exclusion chromatography (SEC) data were collected on a TOSOH Bioscience EcoSEC SEC equipped with a Bryce-type double path refractive index detector and two TSKgel SuperMultiporeHZ-M columns. For the molecular weight and dispersity characterizations, tetrahydrofuran at 40 °C was used as the mobile phase at a flow rate of 0.35 mL/min. The masses of PS standards for calibration range from 500 to 2,890,000 g mol⁻¹. Thermogravimetric analysis (TGA) measurements were performed using a NETZSCH TG 209F3 TGA at a heating rate of 20 °C min⁻¹ with nitrogen serving as the purge gas. Differential scanning calorimetry (DSC) measurements were performed using a TA Q1000 calorimeter at a heating rate of 10 °C min⁻¹ with nitrogen as the purge gas. Ultra-sonication treatment of polymer crystals was conducted using QSonica Q700 Sonicator with a 1/2" standard probe. Heat-pressing was conducted using Dulytek Elite DE10K Electric Hybrid Rosin Press. The microwave treatment of the polymer film was conducted using a CEM Discover automatic microwave reactor with constant heating at 95 °C for 30 min. The Raman spectra were collected on a Thermo Scientific DXR2 Raman Microscope with a 633 nm HeNe laser source. The sublimation experiment was conducted with a Buchi Glass Oven B-585. The monomer formation kinetics in depolymerization was monitored by a LaboACE LC-5060 Recycling Preparative HPLC with 250 nm and 400 nm UV detectors. The extrusion of the polymers was conducted with a Xplore MC 5 Micro Compounder with the 5 ml batch volume and 1.75 mm extruder. Tensile stress-strain tests were conducted using Mechanical Testing System (MTS) Criterion Model 43. Scanning electron microscopy (SEM) was conducted using Quanta 3D FEG. Nanoindentation experiments were performed using a KLA G200 Nano-indenter in an argon filled glovebox. Modulus and hardness values were obtained as the average value for tip penetration of 2500 - 3000 nm. Thin film out-of-plane XRD data were acquired with a Rigaku Smart Lab using a Cu K α source (λ = 1.54 Å). Single crystals were analyzed using a Bruker Quest diffractometer with kappa geometry, an I- μ -S microsource X-ray tube (Cu K α radiation, λ = 1.54178 Å), laterally graded multilayer (Goebel) mirror for monochromatization, and a Photon2 CMOS area detector.

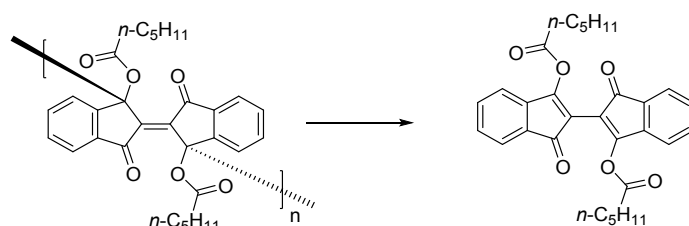
Density Functional Theory (DFT) Calculations

Density function theory calculations were used to characterize the bond energies. These were calculated as the enthalpy changes (i.e., including zero-point energy, and thermal

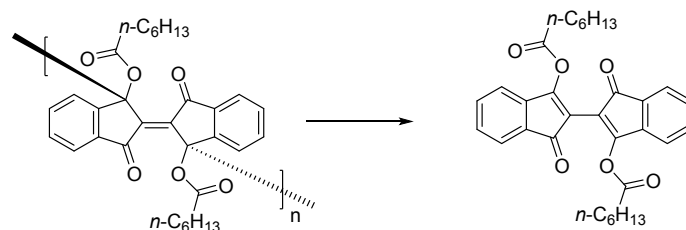
contributions at ambient conditions to the energy difference) upon bond cleavage: $D^\circ(R-X) = \Delta H(R) + \Delta H(X) - \Delta H(RX)^{60}$. All DFT calculations were performed at the ω B97X-D3⁶¹/def2-TZVP⁶² level of theory using ORCA 4.1.2⁶³. The split-RI-J approximation for the Coulomb integrals and chain-of-sphere integration for the Hartree-Fock exchange integrals were used for the frequency calculations⁶⁴. These calculations were performed on dimers of each polymer, with the relevant bond-cleavages performed on the center-most instance of each bond to generate radicals of identical or similar size. The initial geometries were taken from the experimental crystal structures, with the alkyl side-chains replaced by methyl groups and the chain ends terminated with hydrogen atoms (Figure S2). The hydrogen atoms associated with the side-chains and chain termination were then relaxed, while constraining all other atoms based on the crystal structure. Bond dissociation energies designated “with crystal” in the Table S1 were calculated with respect to the structure generated in this way. For those designated “with relaxed dimer” in Table S1, the geometry was further relaxed without any constraints to obtain a geometry without imaginary frequencies. For the radicals generated from homolytic bond cleavage, geometry optimizations were performed without any constraints. Radicals generated from single-bond cleavages were characterized in the doublet state, and radicals generated from double-bond cleavages were characterized in the triplet state (i.e., as triplet carbenes). After these geometry optimizations, frequency calculations were performed to characterize the zero-point energy and thermal energy corrections to the enthalpy.

Synthesis and Processing Procedures

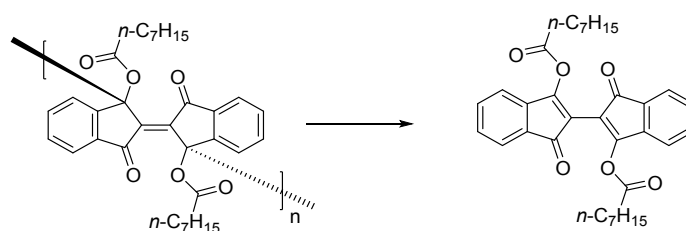
The photopolymerization of monomer crystal BITs³⁵⁻³⁷, MEs³⁸⁻⁴¹ and QDMs⁴² were conducted according to the literature.



Depolymerization of polyBIT-6: In a 25 mL round-bottom flask with a condenser, polyBIT-6 crystals (50.3 mg) were added to 3 mL anisole under argon atmosphere. The mixture was heated at 145 °C for 30 minutes. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH_2Cl_2 as eluent). 49.8 mg of BIT-6 was obtained after evaporation as an orange powder (99% yield).

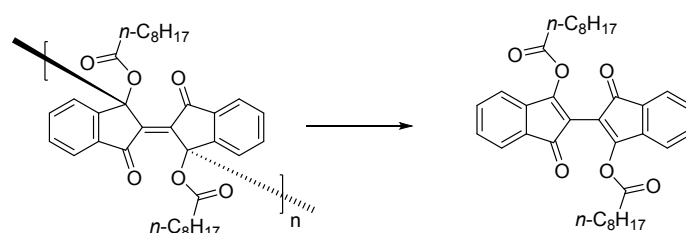


Depolymerization of polyBIT-7: In a 25 mL round-bottom flask with a condenser, polyBIT-7 crystals (58.6 mg) were added to 3 mL anisole under argon atmosphere. The mixture was heated at 145 °C for 30 minutes. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH₂Cl₂ as eluent). 57.8 mg of BIT-7 was obtained after evaporation as an orange powder (99% yield).

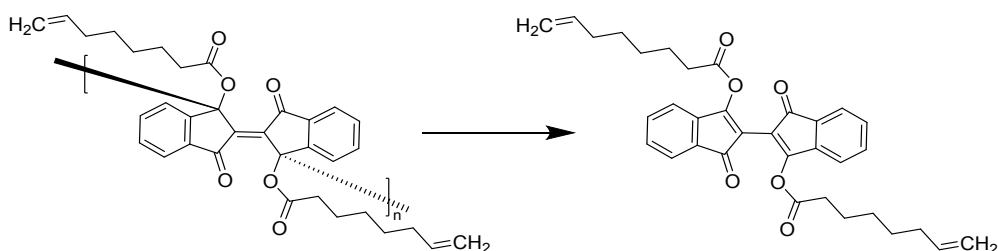


Depolymerization of polyBIT-8: In a 25 mL round-bottom flask with a condenser, polyBIT-8 crystals (99.6 mg) were added to 5 mL anisole under argon atmosphere. The mixture was heated at 145 °C for 30 minutes. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH₂Cl₂ as eluent). 99.5 mg of BIT-8 was obtained after evaporation as an orange powder (>99% yield).

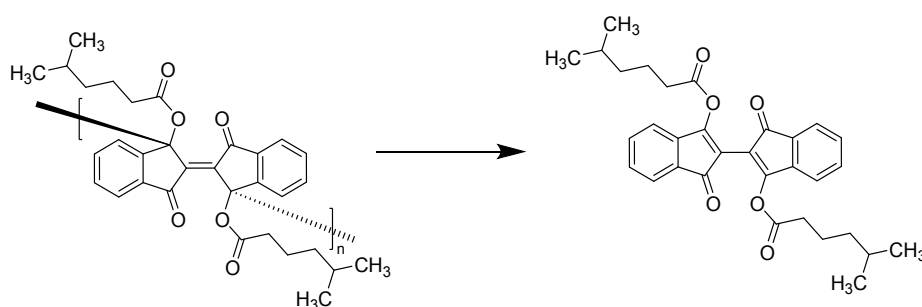
The depolymerization reaction of the pristine thin film (39.4 mg) was conducted with the same method as polyBIT-8 crystals. 31.8 mg of BIT-8 were obtained (98% yield).



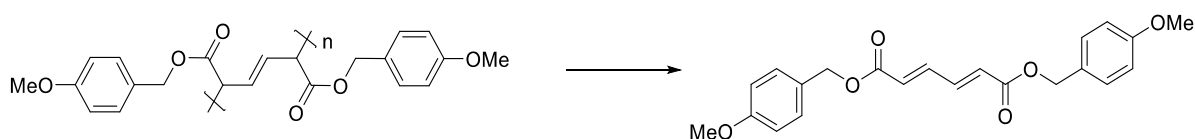
Depolymerization of polyBIT-9: In a 25 mL round-bottom flask with a condenser, polyBIT-9 crystals (47.8 mg) were added to 5 mL anisole under argon atmosphere. The mixture was heated at 145 °C for 30 minutes. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH₂Cl₂ as eluent). 47.3 mg of BIT-9 was obtained after evaporation as an orange powder (99% yield).



Depolymerization of polyBIT-8D: In a 25 mL round-bottom flask with a condenser, polyBIT-8D crystals (40.5 mg) were added to 3 mL anisole under argon atmosphere. The mixture was heated at 155 °C for 1 h. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH₂Cl₂ as eluent). 39.9 mg of BIT-8D was obtained after evaporation as an orange powder (98.5% yield).



Depolymerization of polyBIT-7B: In a 25 mL round-bottom flask with a condenser, polyBIT-7B crystals (45.0 mg) were added to 3 mL anisole under argon atmosphere. The mixture was heated at 135 °C for 30 minutes. Polymer crystals dissolved, and the solution color changed from colorless to orange. After cooling down, anisole was removed from the resulting mixture by a short silica column (CH₂Cl₂ as eluent). 44.5 mg of BIT-7B was obtained after evaporation as an orange powder (99% yield).

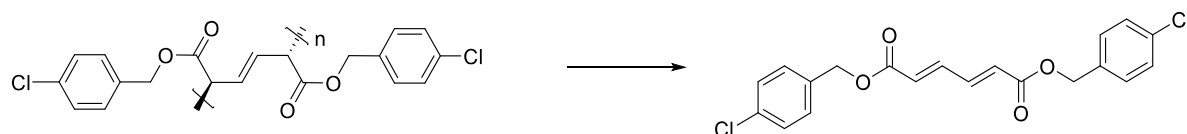


Depolymerization of polyME-OMeB: polyME-OMeB crystals (50.2 mg) were mixed with diphenyl ether (20 mL) in a 50 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux (~258 °C) for 20 min. The reaction setup was cooled down quickly by water (to prevent from possible thermal polymerization-depolymerization equilibrium). The solvent diphenyl ether was removed by a short silica column (CH₂Cl₂:Ethyl Acetate = 15:1) to obtain crude ME-CB with *EE* (major), *EZ* and *ZZ* isomers (86% crude yield, all isomers are polymerizable). The I₂ treatment was applied as a catalyst to facilitate thermal equilibrium reaction of *EE*/*EZ*/*ZZ* isomers. Crude ME-OMeB isomers were mixed with 0.6 mg I₂ (0.02 eq) in 5 mL THF, stirred at room temperature under argon atmosphere overnight, then evaporated under vacuum. The residue

was purified by silica column chromatography (CH_2Cl_2) to give 37.2 mg of pure (*EE*)-ME-OMeB as a white powder (41.0 mg, 82% yield). The *EZ* isomer was too little to be collected. ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 7.34 – 7.28 (m, $\text{CH}=\text{CHCO}_2\text{R}$ and C_6H_4 , 6H), 6.92 – 6.87 (m, C_6H_4 , 4H), 6.25 – 6.14 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 5.14 (s, OCH_2 , 4H), 3.81 (s, CH_3 , 6H).

The depolymerization was also occur at lower temperature in a boiling 2:1 solvent mixture of diphenyl ether and trichlorobenzene ($\sim 228^\circ\text{C}$), but with longer reaction time of 3 h, affording 60% recovery yield (60 mg) of ME-OMeB monomers.

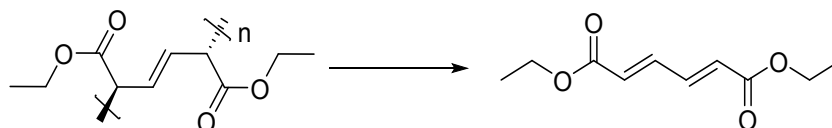
The depolymerization of the sonication-processed film before heat-pressing and after heat pressing were conducted with the same method as polyME-OMeB crystals with recovery yields of 83% and 73%, respectively. The depolymerization reaction of the hot-solvent processed polymer (50.0 mg) was conducted with the same method as polyME-OMeB crystals. 35.9 mg of ME-OMeB was obtained (72% yield).



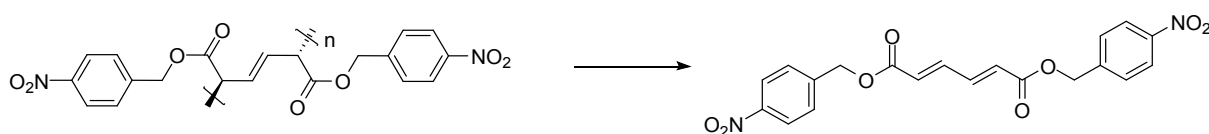
Depolymerization of polyME-CB: polyME-CB crystals (100 mg) were mixed with diphenyl ether (15 mL) in a 50 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux ($\sim 258^\circ\text{C}$) for 20 min. The reaction setup was cooled down quickly by water (to prevent from possible thermal polymerization-depolymerization equilibrium). The solvent diphenyl ether was removed by a short silica column (CH_2Cl_2 :Ethyl Acetate = 15:1) to obtain crude ME-CB with *EE* (major), *EZ* and *ZZ* isomers (95% crude yield). Crude ME-CB isomers were mixed with 1.3 mg I_2 (0.02 eq) in 5 mL THF, stirred at 60°C under argon atmosphere overnight, then evaporated under vacuum. The residue was purified by silica column chromatography (CH_2Cl_2) to give 85 mg of pure (*EE*)-ME-CB. and 6 mg of (*EZ*)-ME-CB as white powders (91% yield in total). (*EE*)-ME-CB: ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 7.38 – 7.28 (m, $\text{CH}=\text{CHCO}_2\text{R}$ and C_6H_4 , 10H), 6.28 – 6.18 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 5.17 (s, OCH_2 , 4H). (*EZ*)-ME-CB: ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 8.45 (ddd, $J = 15.6, 11.6, 1.1$ Hz, $\text{CH}=\text{CHCO}_2\text{R}$, 1H), 7.37 – 7.29 (m, C_6H_4 , 8H), 6.67 (t, $J = 11.6$ Hz, $\text{CH}=\text{CHCO}_2\text{R}$, 1H), 6.14 (d, $J = 15.6$ Hz, $\text{CH}=\text{CHCO}_2\text{R}$, 1H), 6.00 (d, $J = 11.4$ Hz, $\text{CH}=\text{CHCO}_2\text{R}$, 1H), 5.18 (s, OCH_2 , 2H), 5.16 (s, OCH_2 , 2H).

The depolymerization reaction also occurs at lower temperature in a boiling 2:1 solvent mixture of diphenyl ether and trichlorobenzene ($\sim 228^\circ\text{C}$), but with longer reaction time of 2.5 h, affording 81% recovery yield (80.9 mg) of ME-CB monomers.

The depolymerization of the sonication-processed polyME-CB film before heat-pressing and after heat pressing were conducted with the same method as polyME-OMeB crystals with recovery yields of 93% and 92%, respectively.

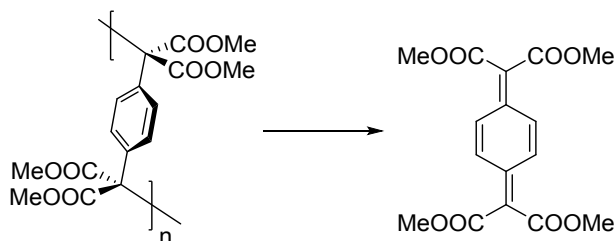


Depolymerization of polyME-Et: polyME-Et crystals (100 mg) were mixed with diphenyl ether (15 mL) in a 50 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux ($\sim 258^\circ\text{C}$) for 20 min. The reaction setup was cooled down quickly by water (to prevent from possible thermal polymerization-depolymerization equilibrium). The solvent diphenyl ether was removed by a short silica column (CH_2Cl_2) to obtain crude ME-Et with *EE* (major), *EZ* and *ZZ* isomers (91% crude yield). Crude ME-CB isomers were mixed with 1.3 mg I_2 (0.02 eq) in 5 mL THF, stirred at 60°C under argon atmosphere overnight, then evaporated under vacuum. The residue was purified by silica column chromatography (CH_2Cl_2) to give 84 mg of pure (*EE*)-ME-Et as white powders (84% yield in total). (*EE*)-ME-Et: ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 7.38 – 7.28 (m, $\text{CH}=\text{CHCO}_2\text{R}$ and C_6H_4 , 10H), 6.28 – 6.18 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 5.17 (s, OCH_2 , 4H). (*EZ*)-ME-CB: ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 7.48 – 7.16 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 6.28 – 6.11 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 4.23 (q, OCH_2 , $J = 7.1$ Hz, 4H), 1.31 (t, CH_3 , $J = 7.1$ Hz, 6H).



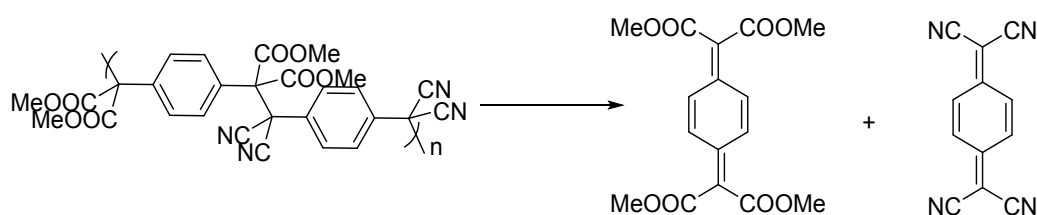
Depolymerization of polyME-NB: polyME-NB crystals (100 mg) were mixed with diphenyl ether (15 mL) and trichlorobenzene (3 mL) in a 50 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux ($\sim 258^\circ\text{C}$) for 20 min.

The reaction setup was cooled down quickly by water (to prevent from possible thermal polymerization-depolymerization equilibrium). The solvent diphenyl ether was removed by a short silica column (CH_2Cl_2 :Ethyl Acetate = 15:1) to obtain crude ME-NB with *EE* (major), *EZ* and *ZZ* isomers (26% crude yield). Crude ME-CB isomers were mixed with 1.3 mg I_2 (0.02 eq) in 5 mL THF, stirred at 60°C under argon atmosphere overnight, then evaporated under vacuum. The residue was purified by silica column chromatography (CH_2Cl_2) to give 22 mg of pure (*EE*)-ME-Et as white powders (22% yield in total). (*EE*)-ME-NB: ^1H NMR (400 MHz, CDCl_3 , ppm) δ : 8.24 (d, $J = 8.7$ Hz, C_6H_4 , 2H), 7.54 (d, $J = 8.9$ Hz, C_6H_4 , 2H), 7.45 – 7.33 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 6.35 – 6.24 (m, $\text{CH}=\text{CHCO}_2\text{R}$, 2H), 5.31 (s, OCH_2 , 4H).

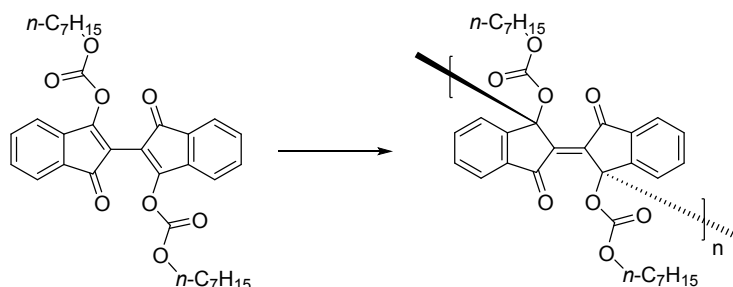


Depolymerization of polyQDM-Me: polyQDM-Me (50.0 mg) were mixed with diphenyl ether (5 mL) in a 25 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux (~258 °C) for 30 min. The reaction mixture was cooled down and purified by a short silica column (CH₂Cl₂:Ethyl Acetate = 10:1) to give 46.4 mg QDM-Me as a light-yellow powder. (92% yield) ¹H NMR (400 MHz, CDCl₃, ppm) δ: 7.43 (s, CH=, 4H), 3.84 (s, OCH₃, 12H).

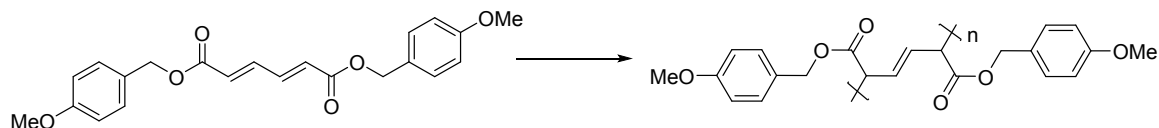
Note, the depolymerization reaction also occurs at lower temperature in boiling trichlorobenzene (~214 °C), but with longer reaction time of 1.5 h, affording 61% recovery yield (26.5 mg) of QDM-Me monomer.



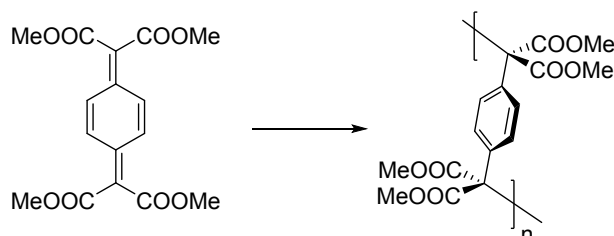
Depolymerization of polyQDM-TCNQ: polyQDM-TCNQ (50.0 mg) were mixed with diphenyl ether (5 mL) in a 25 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux (~258 °C) for 2 hours. The reaction mixture was cooled down and purified by a silica column (CH₂Cl₂:Ethyl Acetate = 10:1) to give 14.1 mg QDM-Me as a light-yellow powder, and 7.0 mg TCNQ as a yellow powder which gradually changes to yellow-green color over time in air. (45% yield, based on QDM-Me) QDM-Me: ¹H NMR (400 MHz, CDCl₃, ppm) δ: 7.43 (s, CH=, 4H), 3.84 (s, OCH₃, 12H). TCNQ: ¹H NMR (400 MHz, CDCl₃, ppm) δ: 7.56 (s, CH=, 4H).



Re-polymerization of recovered BIT-8: The recovered BIT-8 monomer powder was recrystallized in DCM/MeOH to form needle-like crystals (71% recrystallization yield, dissolved monomer can be recovered). BIT-8 crystals (100 mg) were placed in a glass Petri dish and covered with a piece of glass. The monomer was irradiated under visible-light lamps in the photoreactor for 12 h. The resulting polymer crystals were stirred in 50 mL chloroform at reflux for 12 h to remove any unreacted monomer/oligomer. Filtration provided the polymer polyBIT-8 as off-white needle-like crystals (98% yield). The polymer structure was confirmed by single crystal X-ray crystallography.



Re-polymerization of recovered (E,E)-ME-OMeB: (E,E)-ME-OMeB monomer powder was recrystallized in DCM/hexanes to form plate-like crystals (88% recrystallization yield, dissolved monomer can be recovered). (E,E)-ME-OMeB crystals (100 mg) was placed in a glass Petri dish and covered with a piece of glass. The monomer was irradiated under UVA lamps (350 nm) in the photoreactor for 72 h. The resulting polymer crystals were stirred in 50 mL chloroform at reflux for 12 h to remove any unreacted monomer/oligomer. Filtration provided the polymer polyME-OMeB as white plate-like crystals (98% yield). The polymer structure was confirmed by single crystal X-ray crystallography.



Re-polymerization of recovered QDM-Me: QDM-Me monomer powder was recrystallized in ethyl acetate/hexanes to form prism-like crystals (80% recrystallization yield, dissolved monomer can be recovered). crystals (100 mg) were placed in a glass Petri dish and covered with a piece of glass. The monomer was irradiated under UVA lamps (350 nm) in N₂ atmosphere for 12 h. The resulting polymer crystals were stirred in 50 mL chloroform at reflux for 12 h to remove any unreacted monomer/oligomer. The orange polymer crystals were dispersed into smaller particles during the chloroform purification. Filtration gave the polymer polyQDM-Me as an ivory powder (87 mg, 87%). The polymer structure was confirmed by single crystal X-ray crystallography using crude orange polymer crystals.

Determination of Depolymerization Kinetics

20 mg polymer was dispersed in 6 mL of DPE and heated in an oil bath or a sand bath for a specific time. For polyBITs, the polymers were heated at 150 for 2.5, 5, 10, 15, 20, 25 and 30 min, respectively. For polyMEs, the polymers were heated at 240 for 10, 20, 30, 40, 50 and 60 min, respectively. For polyQDMs, the polymers were heated at 260 for 2.5, 5, 10, 15, 20, 25 and 30 min, respectively. The reaction mixture was cooled in a water bath, diluted with 6 mL chloroform, and filtered with a syringe filter. 2 mL of the resulting solution was injected into the LaboACE LC-5060 HPLC to estimate the monomer conversion. BIT monomers and ME monomer peaks were observed with a 250 nm detector, while QDM monomers were observed with a 400 nm detector. The monomer conversion was calculated by monomer peak signal areas divided by reference monomer peak signal areas.

Film fabrication of polymer crystals via ultra-sonication processing.

PolyME-OMeB film: polyME-OMeB crystals (200 mg) were mixed with acetone (35 mL) in a 50 mL beaker. The ultra-sonication probe was then immersed into the solvent. Ultra-sonication parameters were set as follows: amplitude 20, 4 sec pulse and 1 sec rest. The mixture was cooled by an ice-salt bath during the entire 6 h ultra-sonication processing. The resulting white suspension was divided equally into four portions. Filtration of each portion with a vacuum filtration apparatus and a nylon membrane filter provided a free-standing, flexible white polymer film (around 50 mg each), with quantitative overall yield.

PolyME-CB film: polyME-CB crystals (200 mg) were mixed with acetone (35 mL) in a 50 mL beaker. The ultra-sonication probe was then immersed into the solvent. Ultra-sonication parameters were set as follows: amplitude 20, 4 sec pulse and 1 sec rest. The mixture was cooled by an ice-salt bath during the entire 4 h ultra-sonication processing. The resulting off-white suspension was divided equally into four portions. Filtration of each portion with a vacuum filtration apparatus and a nylon membrane filter provided a free-standing, flexible white polymer film (around 50 mg each), with quantitative overall yield.

PolyBIT-8 film: polyBIT-8 crystals (200 mg) were mixed with acetone (35 mL) in a 50 mL beaker. The ultra-sonication probe was then immersed into the solvent. Ultra-sonication parameters were set as follows: amplitude 15, 4 sec pulse and 1 sec rest. The mixture was cooled by an ice-salt bath during the entire 1 h ultra-sonication processing. Longer ultra-sonication time would lead to a more brittle film. The resulting pale-yellow suspension was divided equally into four portions. Filtration of each portion with a vacuum filtration apparatus and a nylon membrane filter provided a free-standing, flexible, pale-yellow polymer film (around 50 mg each), with quantitative overall yield.

Heat-pressing of the polymer thin films.

PolyME-OMeB sonicated-processed film: One piece of polyME-OMeB pristine film was placed between two pieces of smooth aluminum foil (glossy sides facing the film). This sandwich structure was pressed under around 5 MPa pressure at room temperature for 2.5 min, then heat-pressed under around 5 MPa pressure at 120 °C for 5 min. The aluminum foils were carefully removed, and a semi-transparent and glossy polymer thin film was obtained.

PolyME-OMeB solution-processed film: 100 mg of polyME-OMeB solution-processed solid was placed between two pieces of weighing paper. This sandwich structure was pressed under around 1 MPa pressure at room temperature for 2.5 min, then heat-pressed under

around 1 MPa pressure at 70 °C for 2.5 min. The weighing papers were carefully removed, and a semi-transparent polymer thin film was obtained.

PolyME-CB film: One piece of polyME-CB pristine film was placed between two pieces of smooth aluminum foil. This sandwich structure was pressed under around 5 MPa pressure at room temperature for 2.5 min, then heat-pressed under around 5 MPa pressure at 120 °C for 5 min. The aluminum foils were carefully removed, and a semi-transparent and glossy polymer thin film was obtained.

PolyBIT-8 film: One piece of polyBIT-8 pristine film was placed between two pieces of smooth aluminum foil. This sandwich structure was heat-pressed under around 5 MPa pressure at 50 °C for 2.5 min. The aluminum foils were carefully removed (if they were stuck with the polymer film, acetone could be applied to wet the interface). The resulting polymer film was immersed in acetone for 30 seconds and a glossy pale-yellow polymer-thin film was obtained.

Preparation of the polymer samples for tensile stress-strain tests.

This method applies for both solution-processed films and sonication-processed films. Roughly 25 mm × 5 mm strips were cut from heat-pressed polymer thin films for tensile stress-strain tests. Both ends of the polymer strip were bonded to a piece of polyethylene terephthalate (PET) sheet using epoxy glue for fixtures. The initial length for (engineering) strain calculation was estimated by the distance between two PET sheets, while the cross-sectional area was estimated by width multiplied by thickness of the strip. For testing, both edges of PET sheets should be aligned with edges of the fixtures or be exposed to ensure a legitimate initial length.

Hot-Solvent processing of polyME-OMeB crystals

polyME-OMeB crystals (3.0 g) were mixed with diphenyl ether (3 mL) in a 25 mL round-bottom flask under argon atmosphere. The reaction mixture was heated at 185 °C for 15 min. The resulting viscous polymer solution was cooled down and poured into 100 mL methanol. The sticky polymer residue was left at the bottom of the flask. This polymer (with solvent residues) was dried in a vacuum oven at 80 °C for 24 hours to obtain a white hard polymer solid, which is amorphous measured by powder XRD. (2.8 g, 93%)

Extrusion and 3D printing of solution-processed polyME-OMeB

Hot-solvent processed polyME-OMeB solid (5.0 g) were fed into a microcompounder which was set at 180 °C. The polymer was molten and stable at a setting of 170 °C and 2000 rpm in

the microcompounder. The off-white polymer filament with a diameter of roughly 1.6 mm was extruded and collected. The extruded polymer filament was then fed into the 3D printer to print the “P” shape object. 3D-printing parameters are shown in the section below.

Separation of polyBIT-8 and polyME-CB from a mixture of common polymers.

polyBIT-8 (100.1 mg), polyME-CB (100.5 mg), low-density polyethylene (50.2 mg, $M_n = 7700$, $PDI = 4.5$, Aldrich), polypropylene (51.7 mg, amorphous, Aldrich), polystyrene (61.0 mg, $M_w = 192000$, Aldrich), polycarbonate (48.9 mg, $T_g = 200\text{ }^\circ\text{C}$, PolyK Technologies), polyetherimide (55.1 mg, $T_g = 260\text{ }^\circ\text{C}$, PolyK Technologies) were mixed with diphenyl ether (6 mL) in a 25 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at $160\text{ }^\circ\text{C}$ for 30 min. The reaction setup was cooled down and poured into 100 mL hexanes. Filtration of this mixture gave a filtrate containing the crude BIT-8 monomer and polymer precipitate. Diphenyl ether was removed from the BIT-8 filtrate by a short silica column (CH_2Cl_2 as eluent). 100 mg of BIT-8 was recovered (>99% yield). The polymer precipitate containing polyME-CB, LDPE, PP, PS, PC and PEI were mixed with diphenyl ether (6 mL) in a 25 mL round-bottom flask with a condenser under argon atmosphere. The reaction mixture was heated at reflux ($\sim 258\text{ }^\circ\text{C}$) for 20 min. The reaction setup was cooled down quickly by water (to prevent from possible thermal polymerization-depolymerization equilibrium) and poured into 50 mL hexanes. Filtration of this mixture gave a filtrate containing crude ME-CB monomers and polymer precipitate. Diphenyl ether was removed from the ME-CB filtrate by a short silica column (CH_2Cl_2 :Ethyl Acetate = 15:1). 84.0 mg of ME-CB monomers (with *EE*, *EZ* and *ZZ* isomers) were recovered (84% yield).

60. Hayes, W. M. *CRC Handbook of Chemistry and Physics* (CRC Press, Boca Raton, FL, ed. 97, 2017).
61. Lin, Y.-S. et al. Long-Range Corrected Hybrid Density Functionals with Improved Dispersion Corrections. *J. Chem. Theory Comput.* 2013, 9, 263-272.
62. Weigend, F. & Ahlrichs, R., Balanced basis sets of split valence, triple zeta valence and quadruple zeta valence quality for H to Rn: Design and assessment of accuracy. *Phys. Chem. Chem. Phys.* 2005, 7, 3297-3305.
63. Neese, F. The ORCA program system. *WIREs Comput. Mol. Sci.* 2012, 2, 73-78.

64. Neese, F. et al. Efficient, approximate and parallel Hartree-Fock and hybrid DFT calculations. A 'chain-of-spheres' algorithm for the Hartree-Fock exchange. *Chem. Phys.* 2009, 356, 98-109.

II. Supplementary Figures and Tables

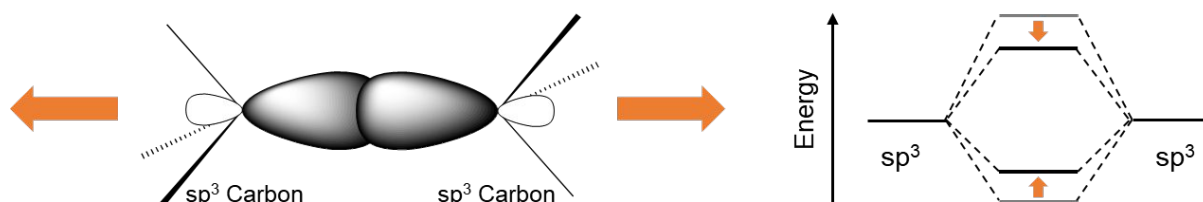


Figure. S1 | Illustration of stretching a C-C bond between two carbon atoms with a sp^3 hybridization and corresponding molecular orbitals.

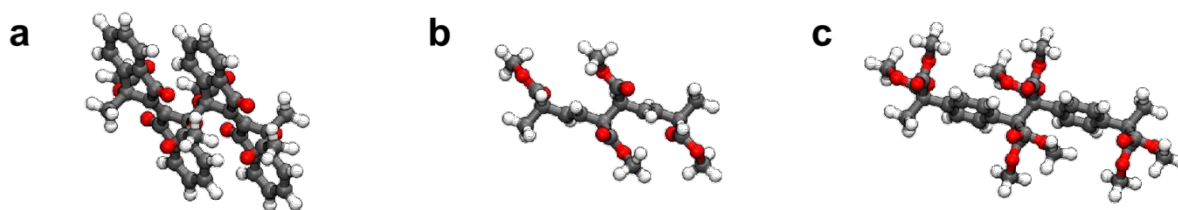


Figure. S2 | Structures of the crystalline polymer dimers used to calculate the bond energies at using DFT for (a) polyBIT, (b) polyME, and (c) polyQDM.

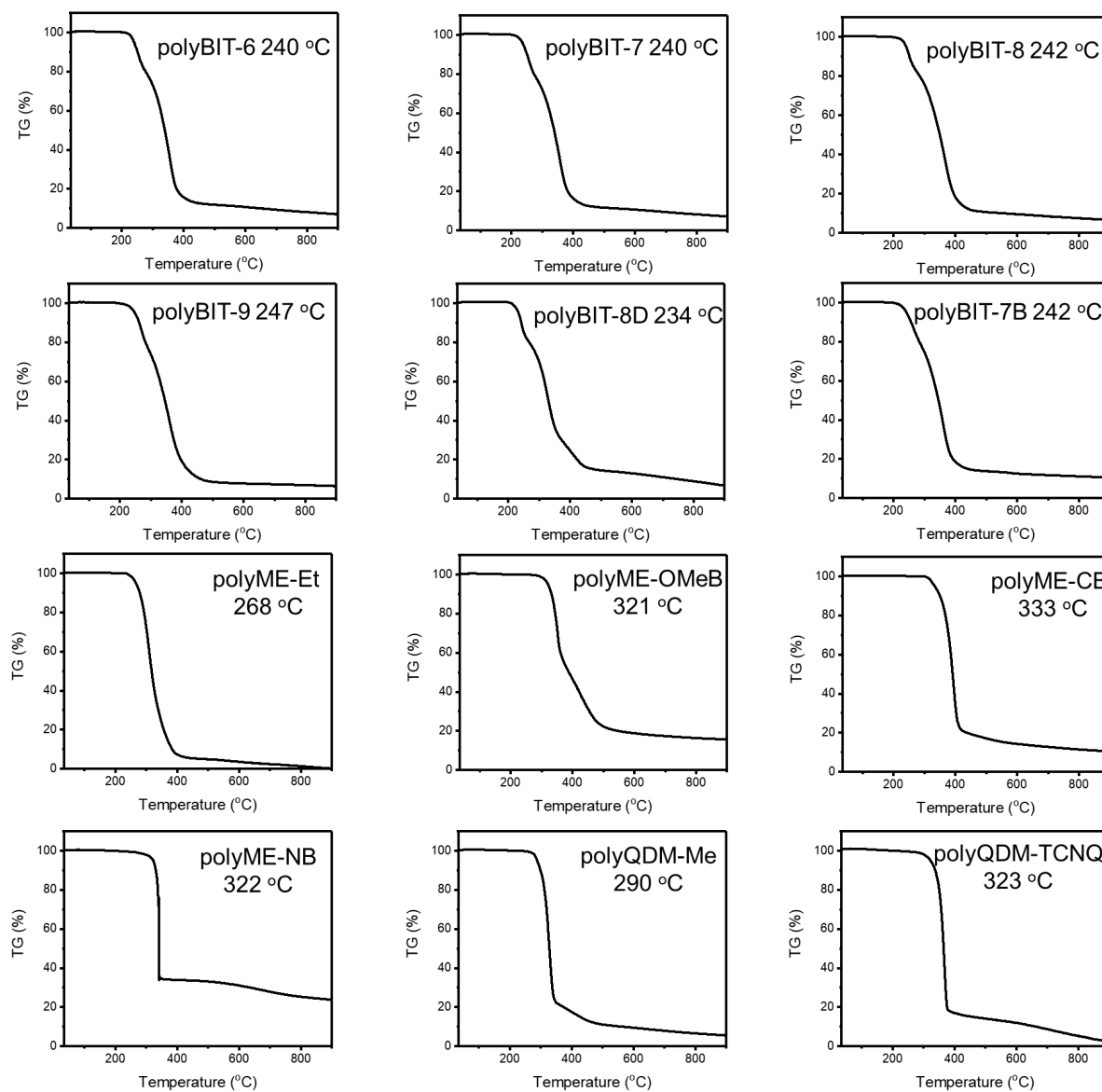


Figure. S3 | Thermogravimetric analysis curves of all 12 polymers. Temperatures at 95% TG are shown beside each polymer name.

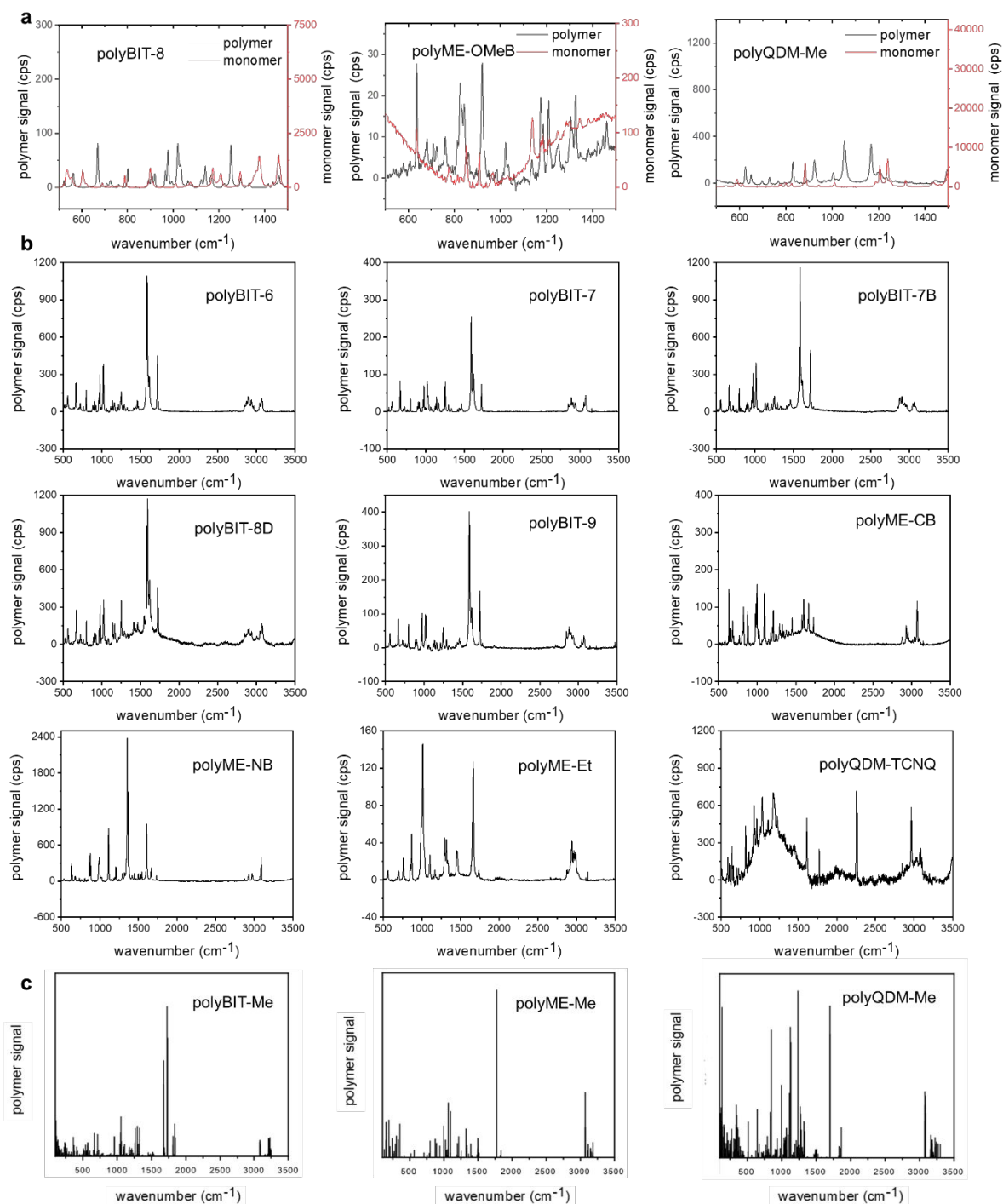


Figure. S4 | **a.** Raman spectra of the selected three representative polymers (polyBIT-8, polyME-OMeB and polyQDM-Me) with their corresponding monomers. Monomer Raman peak positions are significantly different from their polymers. **b.** Raman spectra of the rest nine polymers. **c.** DFT calculated Raman spectra of the model compounds of polyBIT-Me, polyME-Me and polyQDM-Me with methyl group as side chains.

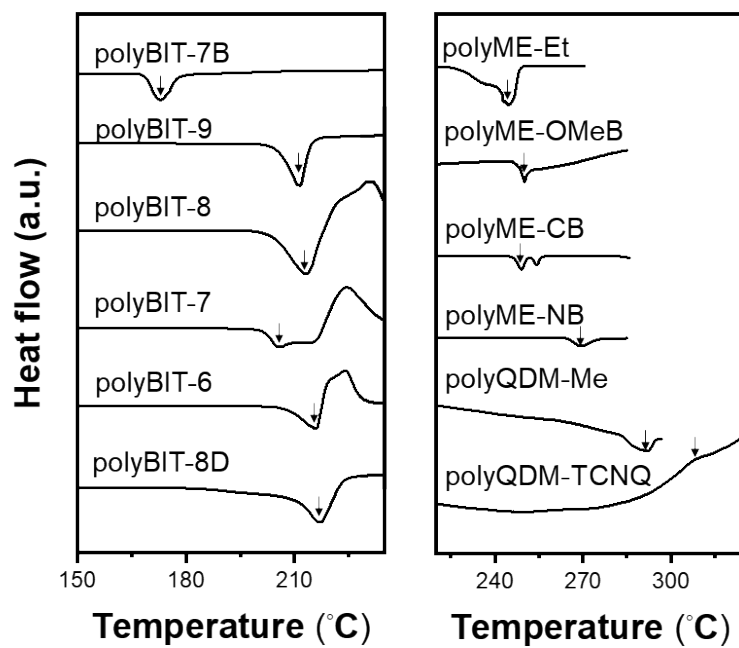


Figure. S5 | DSC profiles of all photopolymerized polymer derivatives (exothermic up). Arrows indicate the first decomposition peak of the polymer.

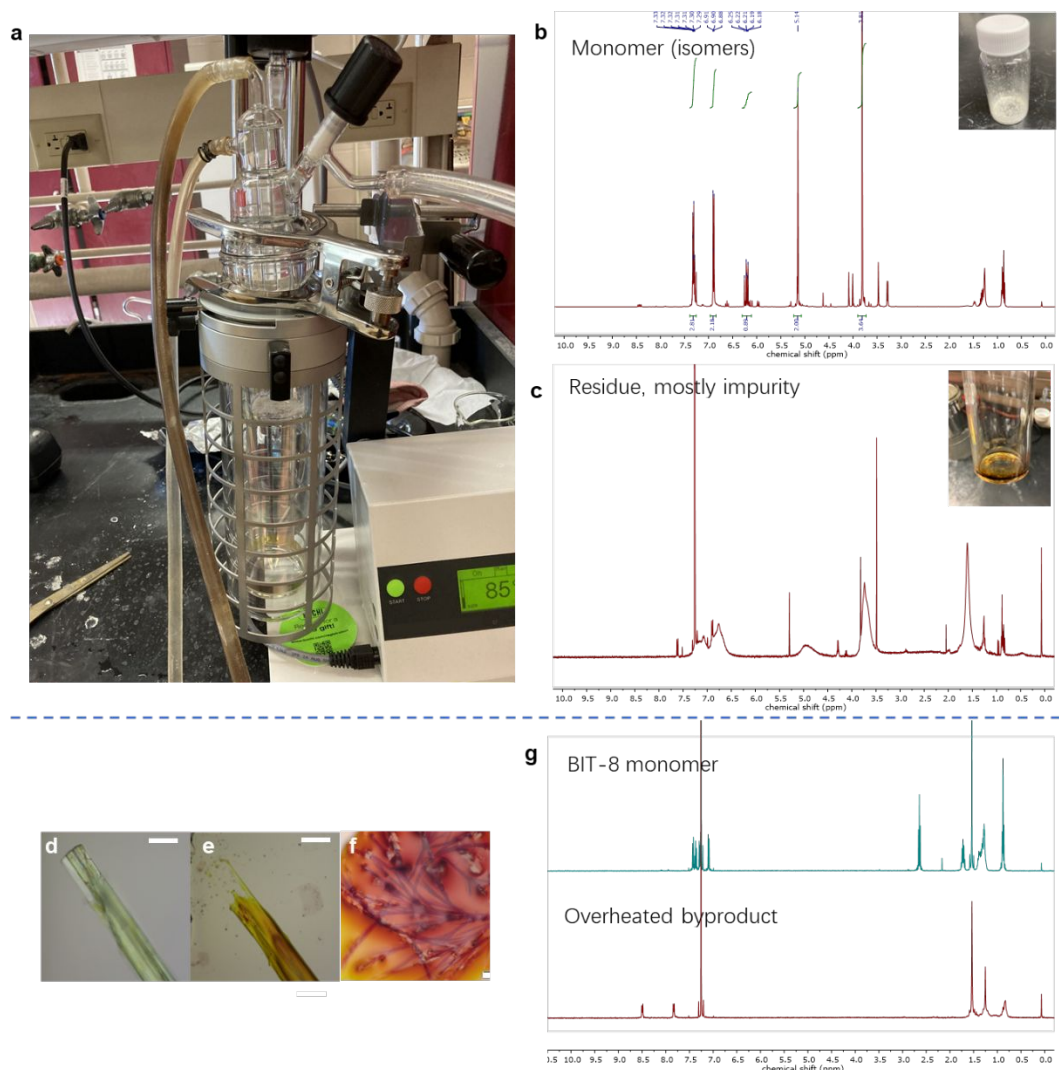


Figure. S6 | The solid-state heating experiment for polyME-OMeB and polyBIT-8. **a.** The sublimation apparatus with polyME-OMeB in a heating glass oven. (100mg, 250 °C, 2 h) **b.** Sublimed ME-OMeB monomers (isomers, 34%) and its ^1H -NMR spectrum. **c.** The non-volatile residue and its ^1H -NMR spectrum. This residue has little polymer. (See the Methods section for ^1H -NMR chemical shifts of pure polyME-OMeB). **d.** Optical image of a polyBIT-8 crystal at room temperature. **e.** Optical image of the polyBIT-8 crystal when reaching 215 °C (under a heating rate of 5°C/min). **f.** Optical image of polyBIT-8 kept at 215 °C until melted, then cooled down to room temperature. **g.** Overlays of ^1H NMR spectra of BIT-8 fresh monomers (top) and the melted yellow byproduct (bottom) (25°C, CDCl_3). These results verify that the endothermic peak observed in DSC profile correspond to depolymerization of pure polyME-OMeB without solvent. However, currently this is not an efficient way to obtain monomers with high yield and purity. Therefore, a solvent-based method was developed in this work to obtain monomers in high yield for all polymers.

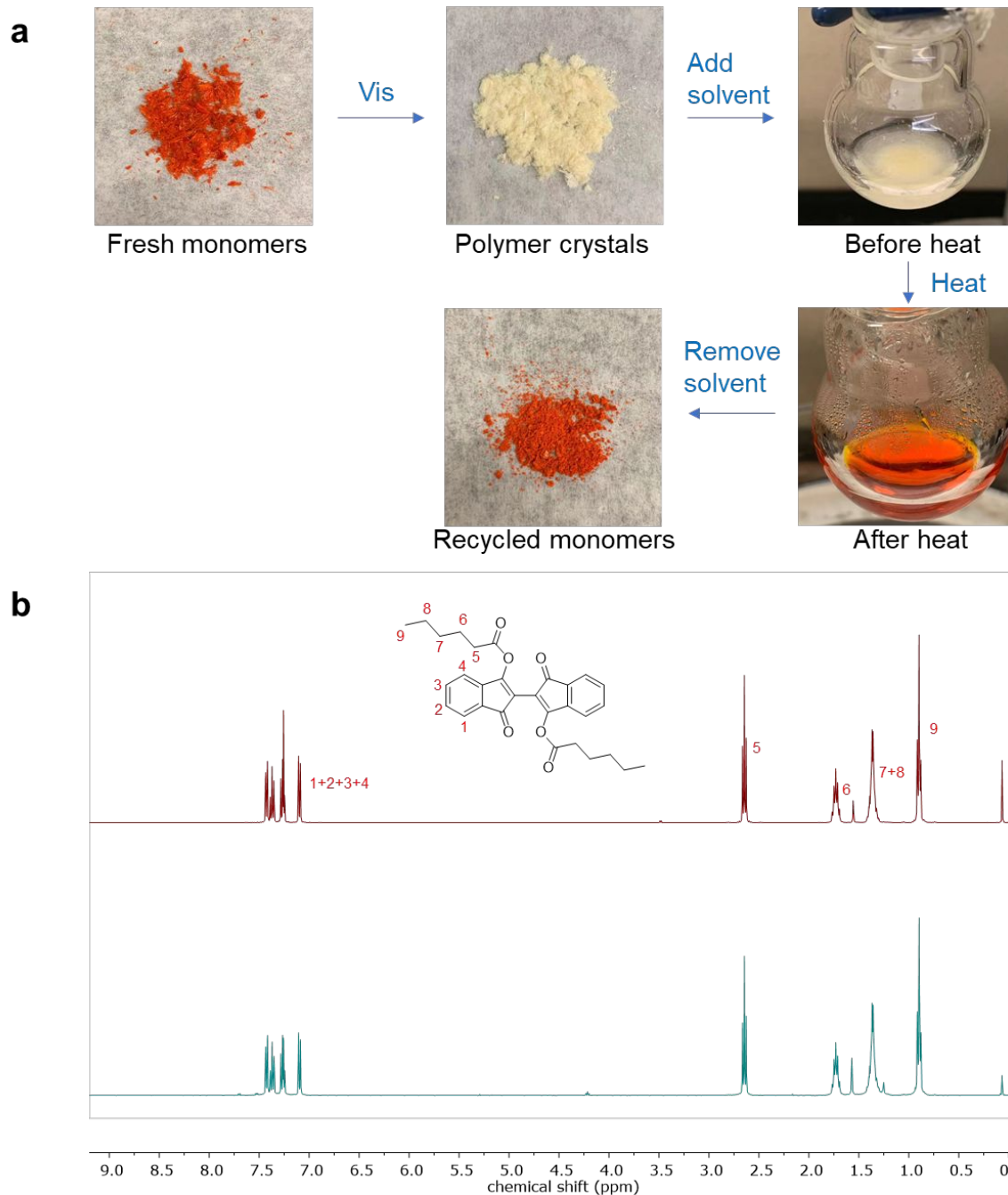


Figure. S7 | a. Photographs of the fresh BIT-6 monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-6 fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C , CDCl_3).

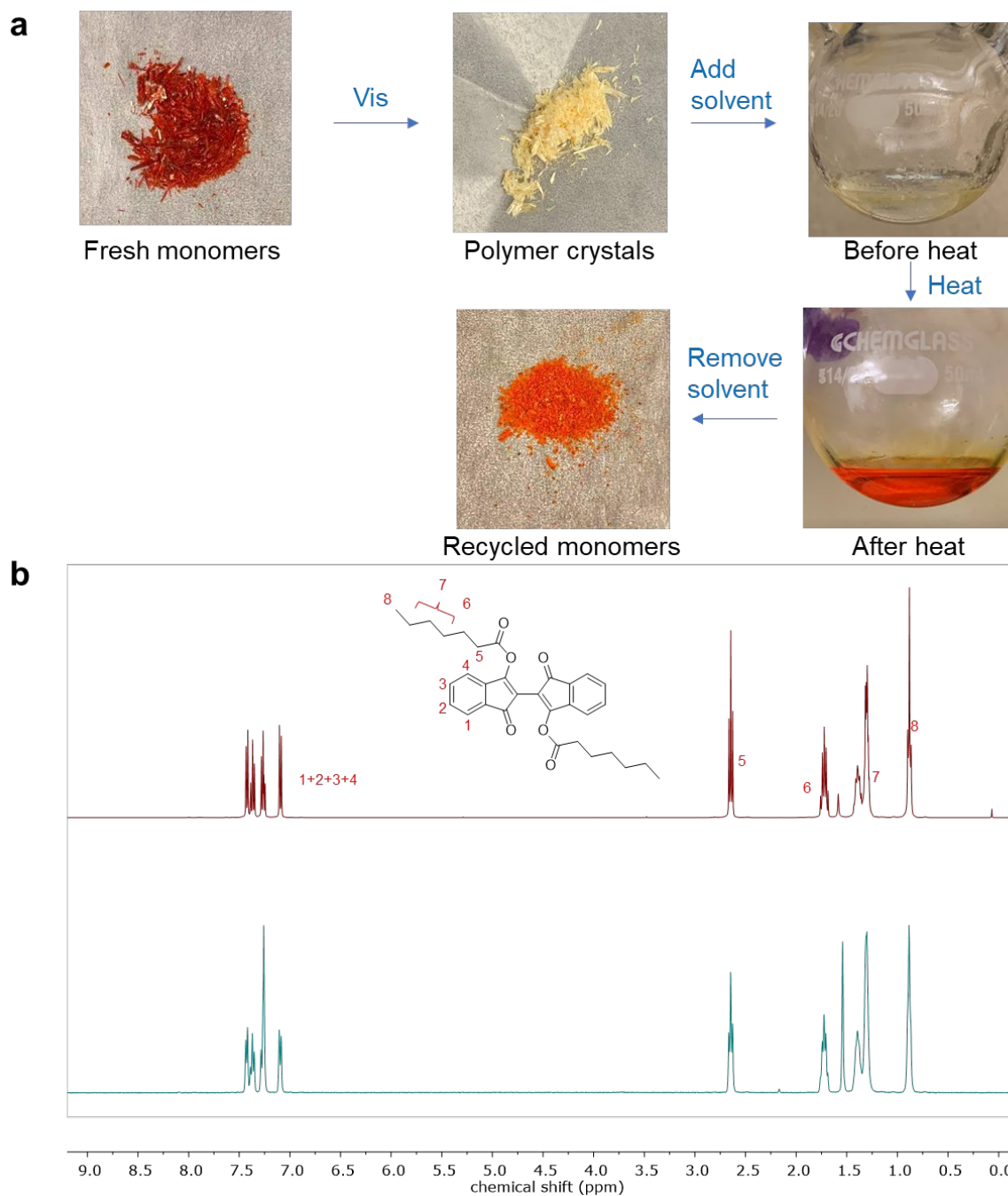


Figure. S8 | **a.** Photographs of the fresh BIT-7 monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-7 fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C , CDCl_3).

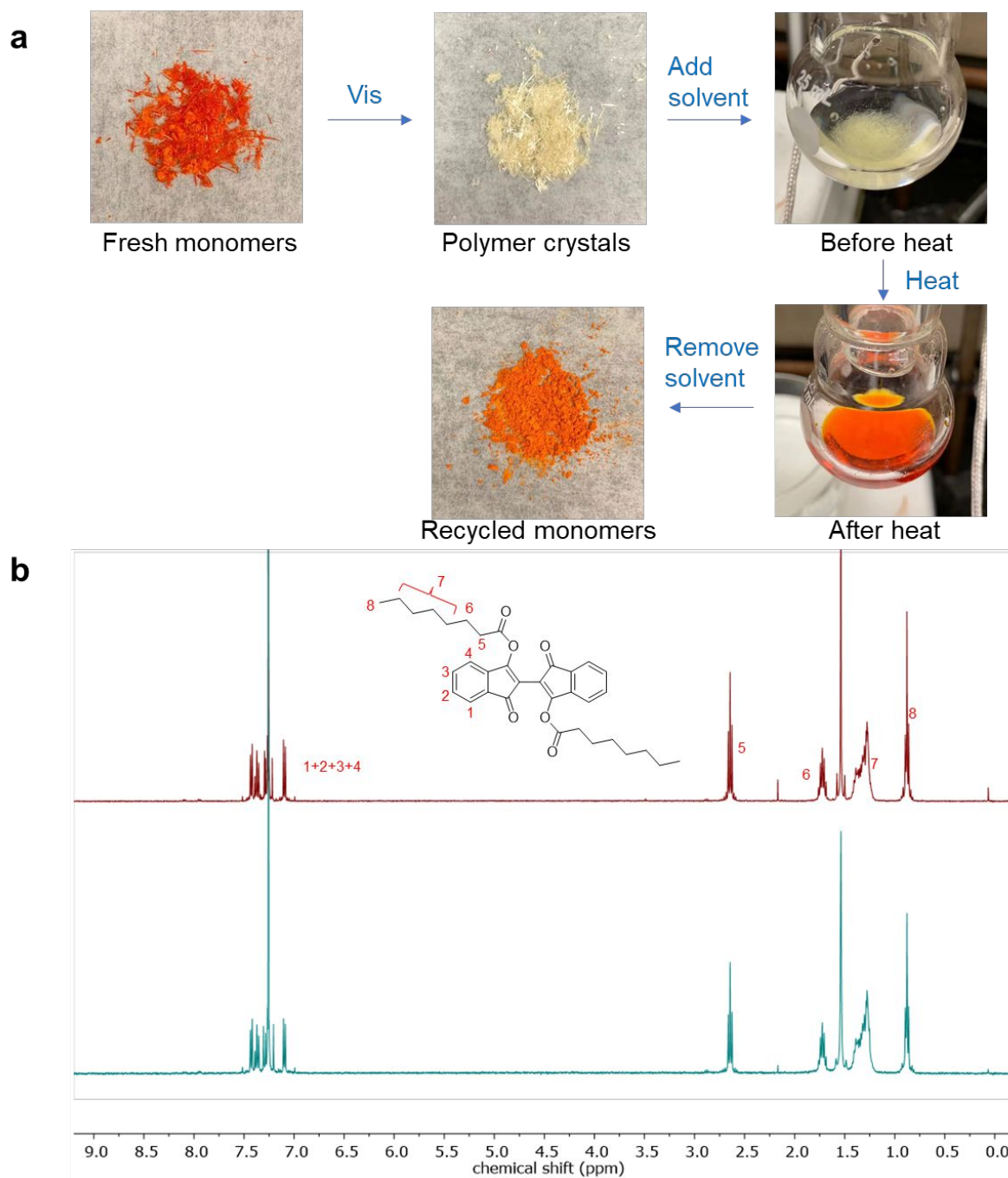


Figure. S9 | **a.** Photographs of the fresh BIT-8 monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-8 fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C , CDCl_3 , with residual solvent peaks at 7.26, 2.17, and 1.56 ppm for CHCl_3 , acetone, and H_2O , respectively)

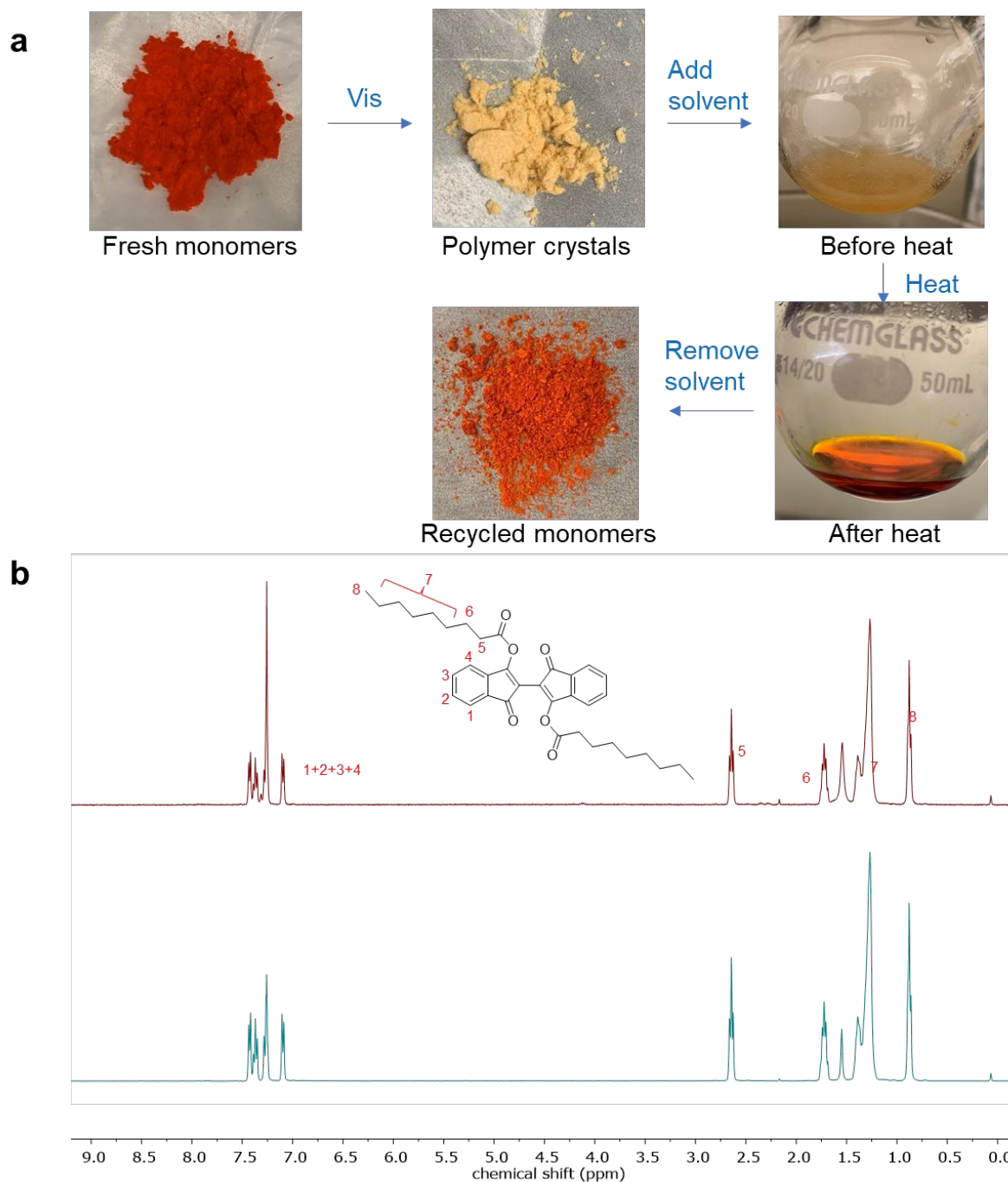


Figure. S10 | a. Photographs of the fresh BIT-9 monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-9 fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C , CDCl_3)

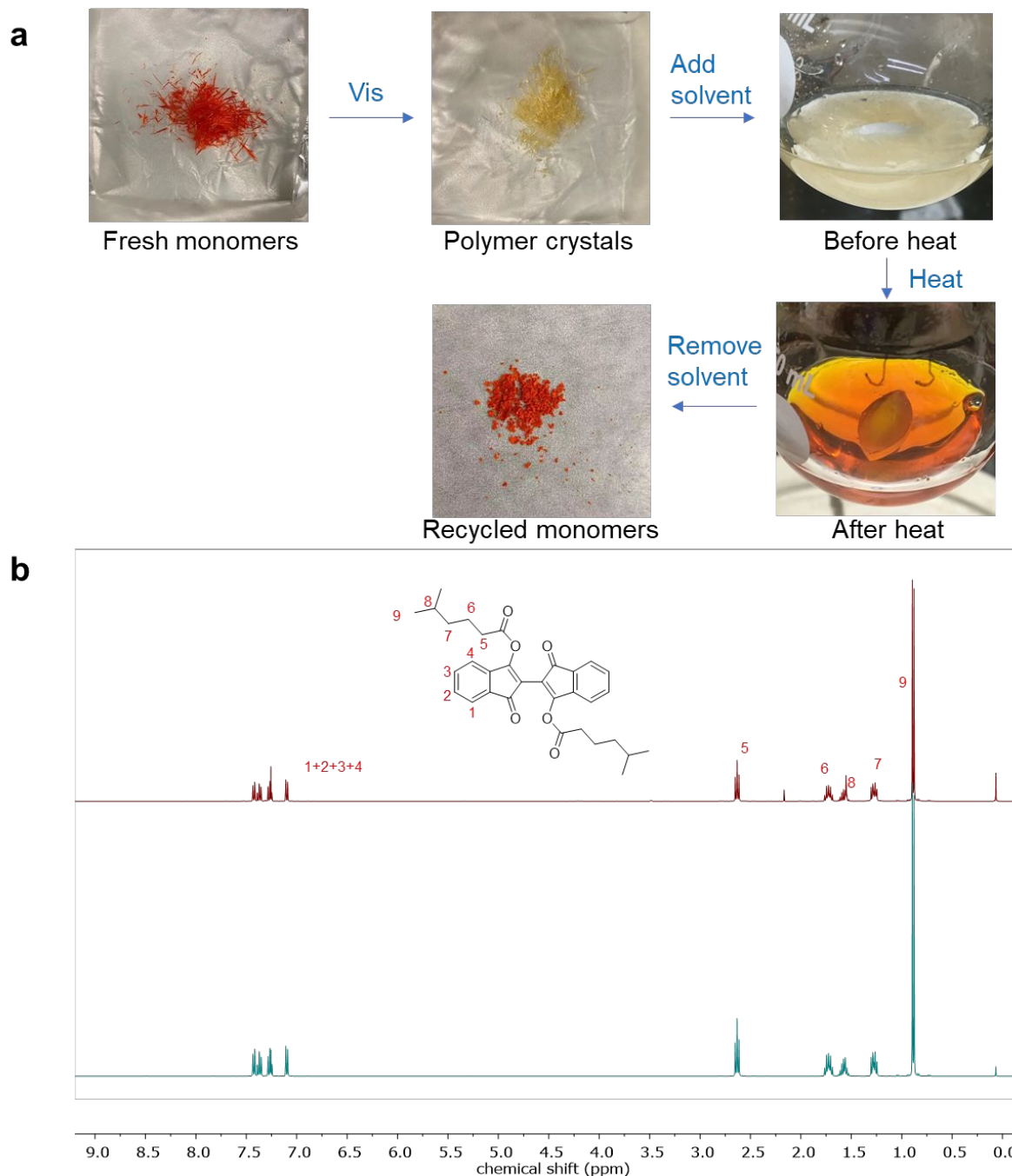


Figure. S11 | a. Photographs of the fresh BIT-7B monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-7B fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C, CDCl_3)

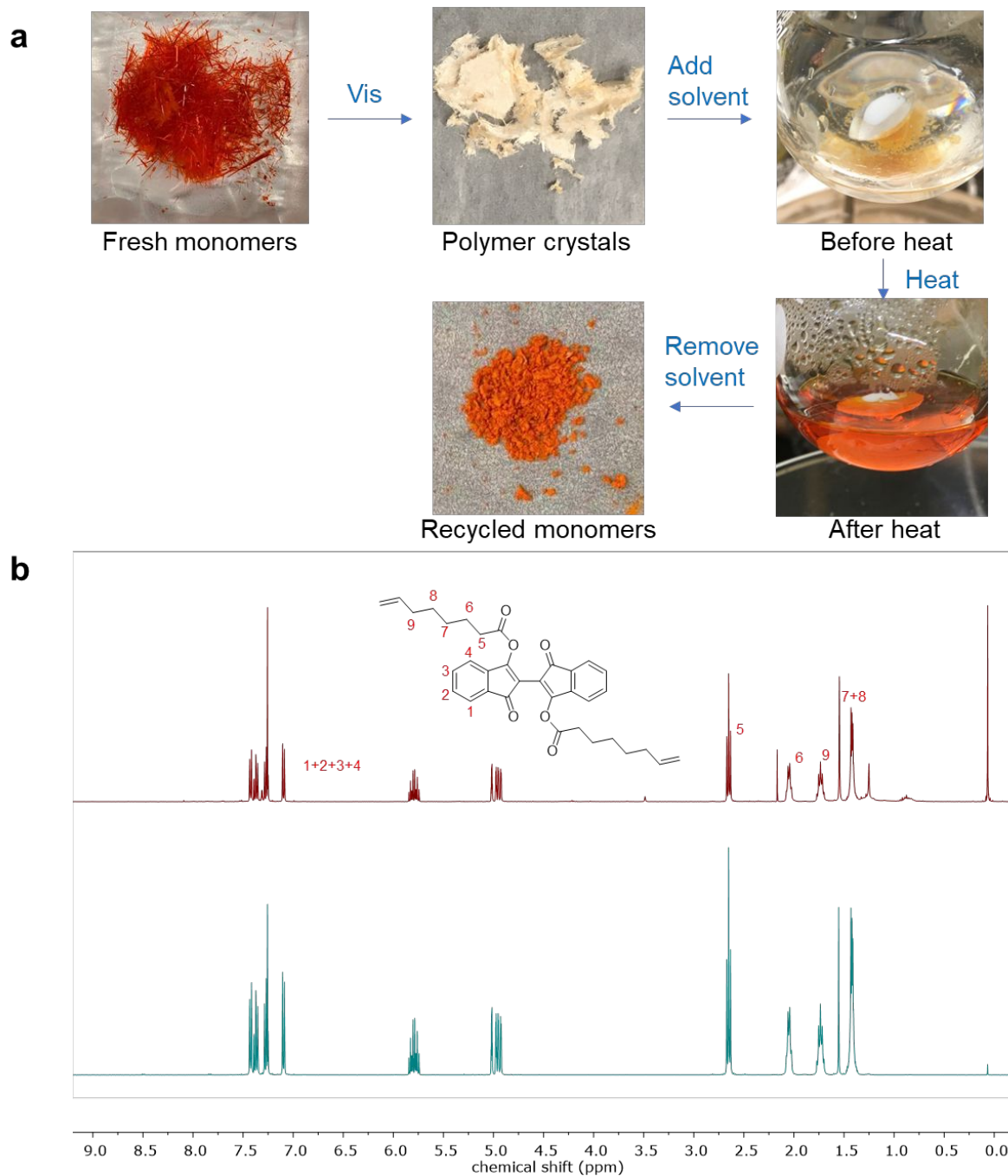


Figure. S12 | a. Photographs of the fresh BIT-8D monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of BIT-8D fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C, CDCl_3)

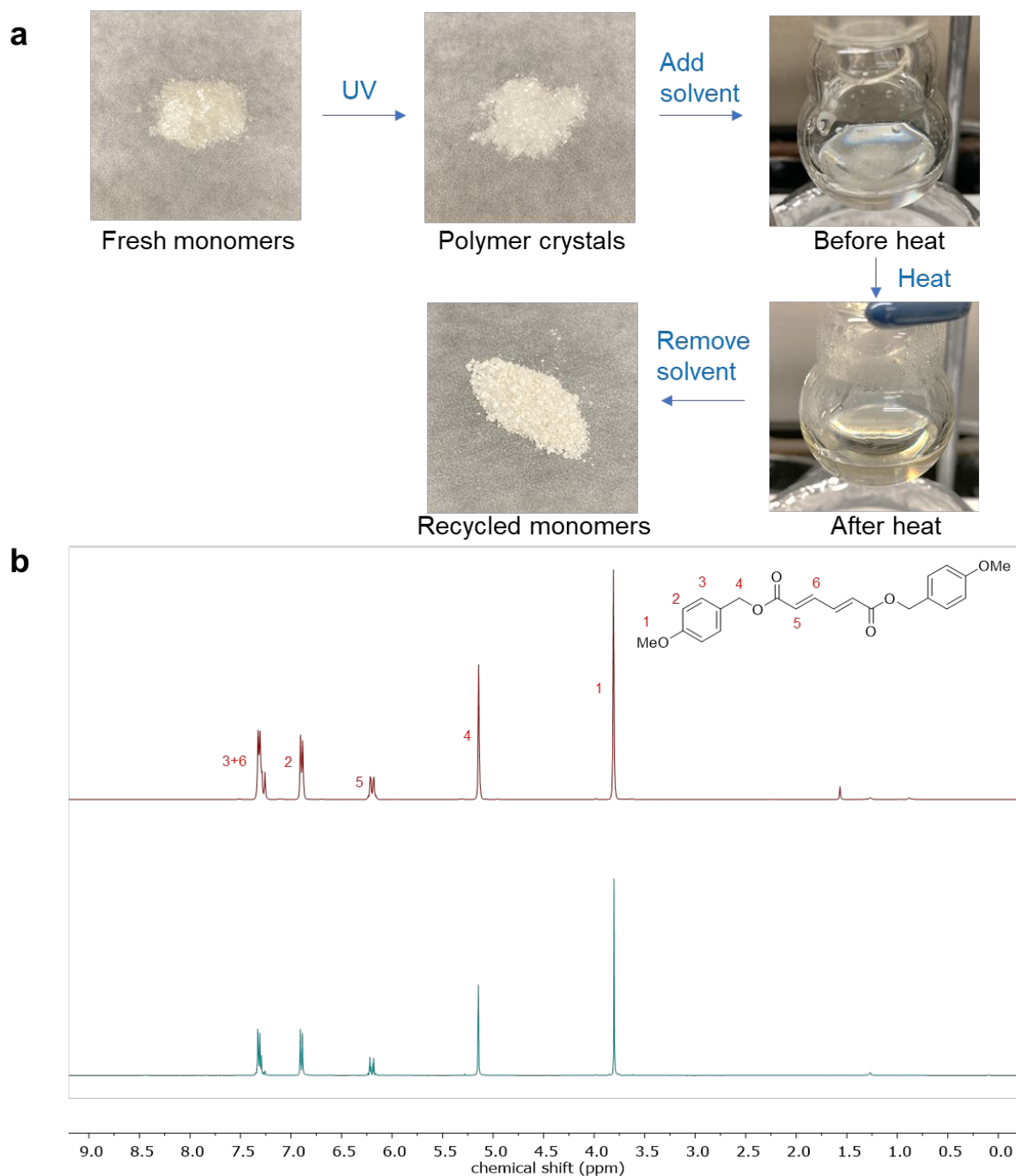


Figure. S13 | a. Photographs of the fresh ME-OMeB monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of ME-OMeB fresh monomers (top) and recycled monomers via solution-processed method (bottom) (25°C, CDCl_3).

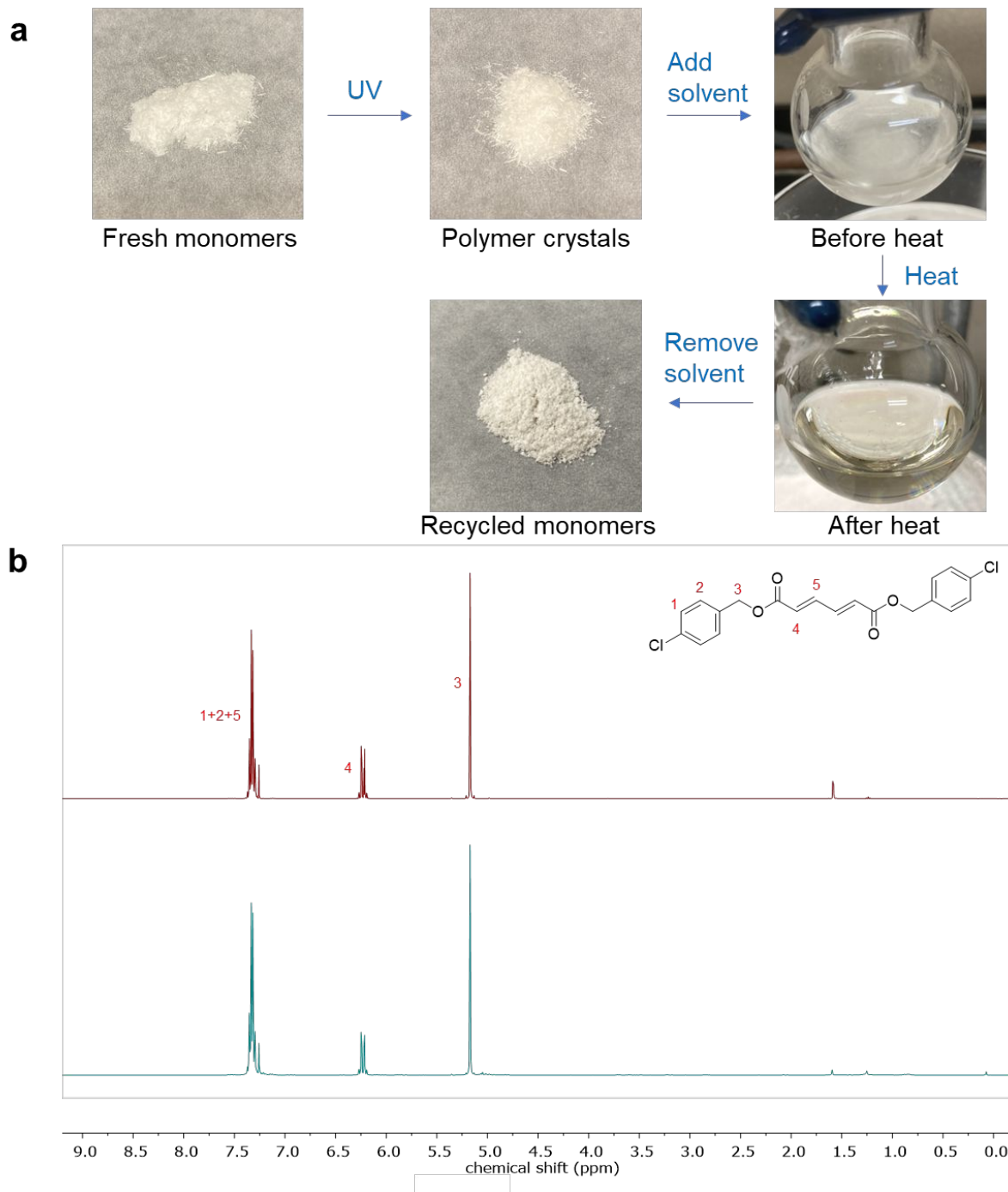


Figure. S14 | a. Photographs of the fresh ME-CB monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of fresh (*EE*)-ME-CB (top) and recycled (*EE*)-ME-CB via solution-processed method (bottom) (25°C, CDCl_3). Note that the polymerizable configuration of ME-CB is the (*ZZ*)- configuration. (*EE*)-ME-CB has the thermodynamically stable configuration, but unlike (*EE*)-ME-OMeB, it is not known to undergo any photopolymerization process yet⁴⁰.

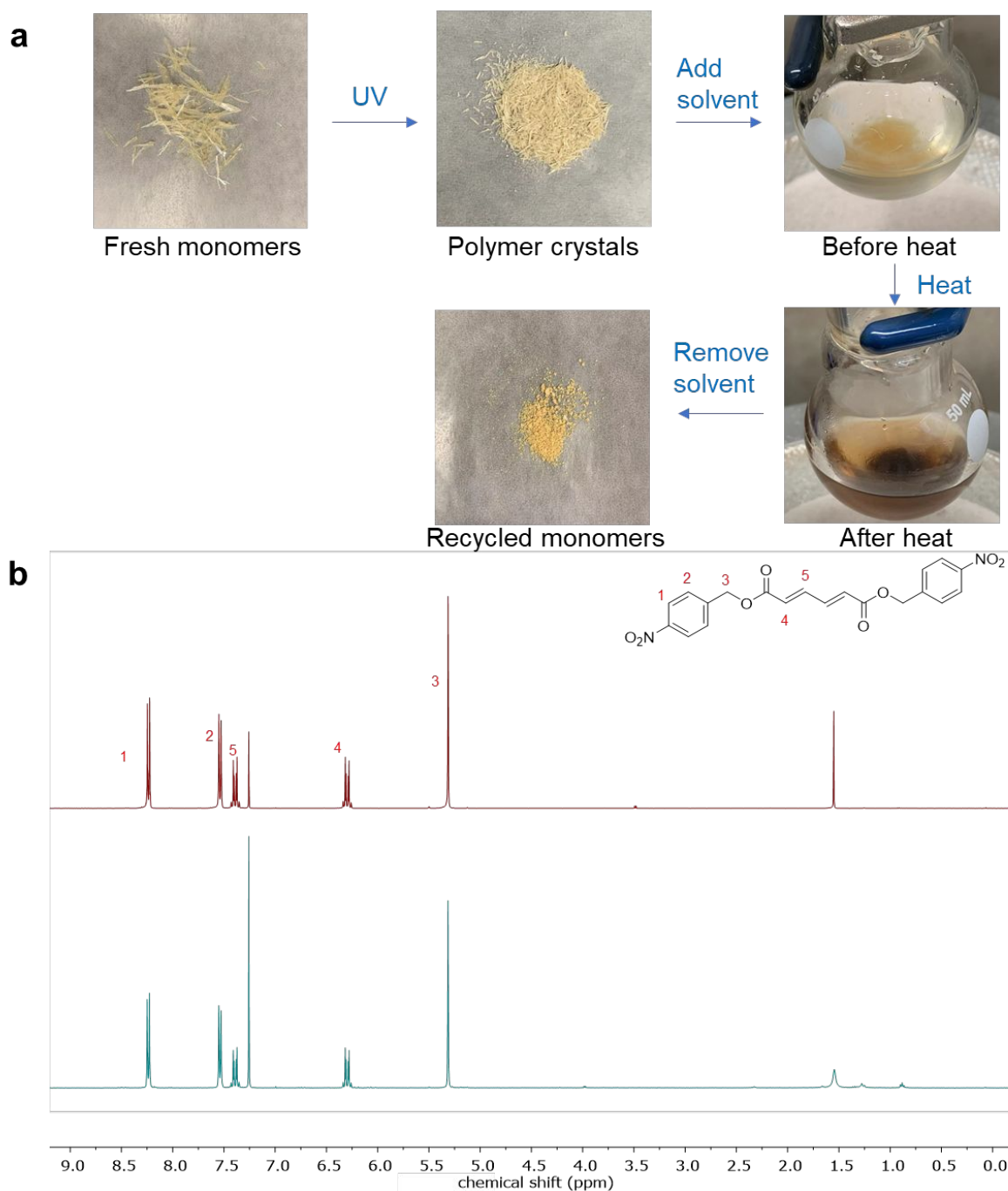


Figure. S15 | a. Photographs of the fresh ME-NB monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of fresh (*EE*)-ME-NB (top) and recycled (*EE*)-ME-NB via solution-processed method (bottom) (25°C , CDCl_3).

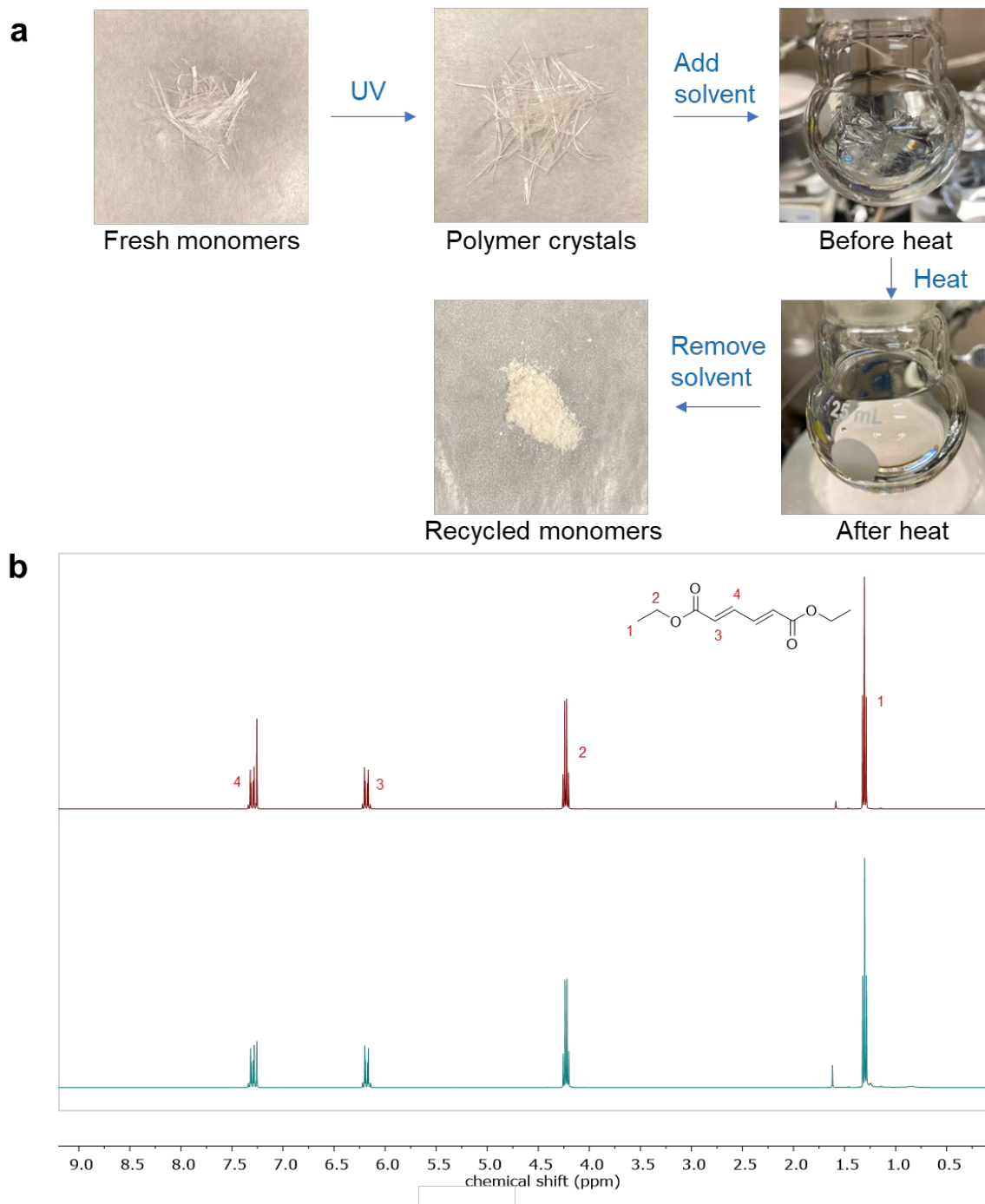


Figure. S16 | **a.** Photographs of the fresh ME-Et monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. **b.** Overlays of ^1H NMR spectra of fresh (*EE*)-ME-Et (top) and recycled (*EE*)-ME-Et via solution-processed method (bottom) (25°C, CDCl_3).

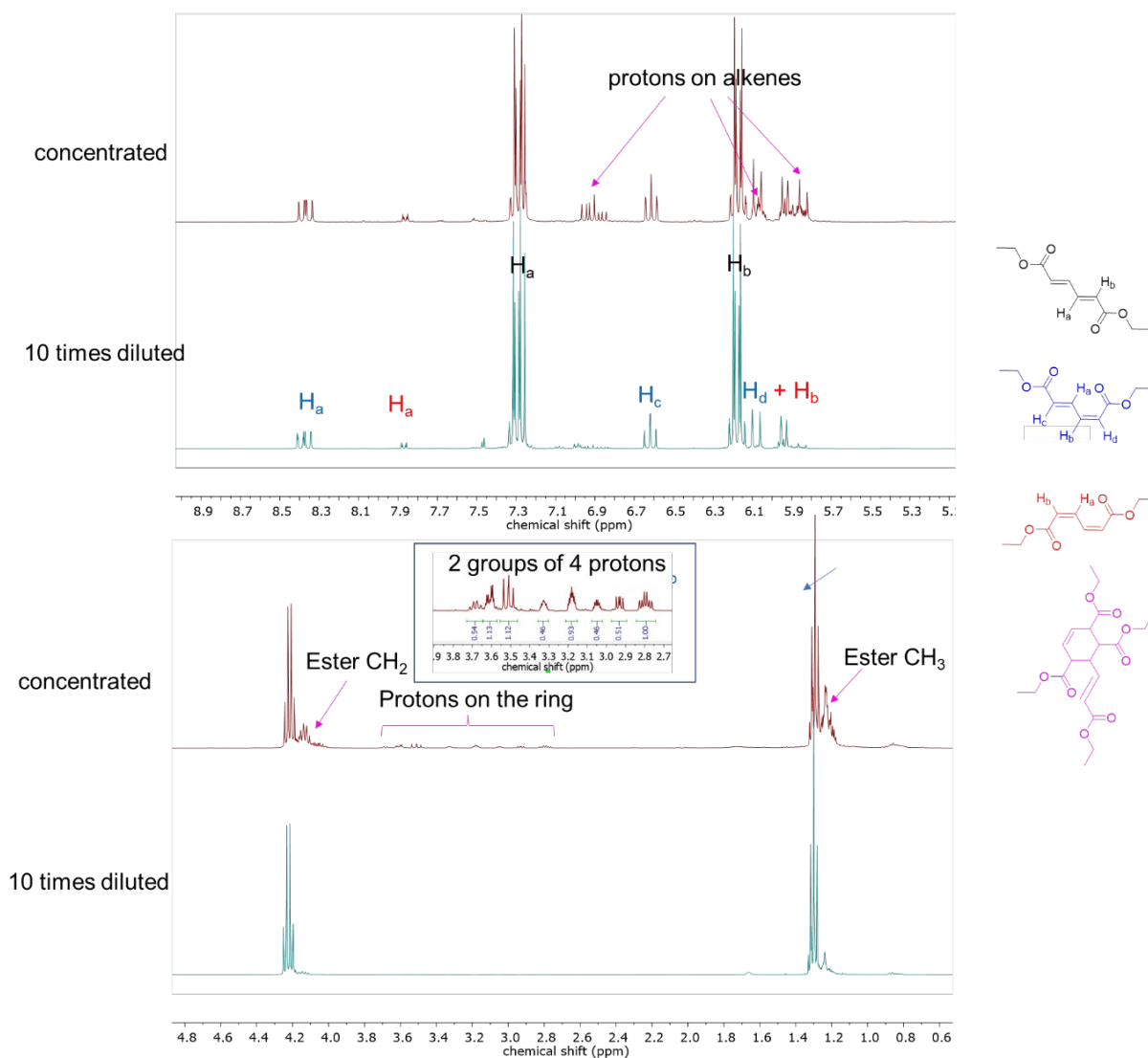
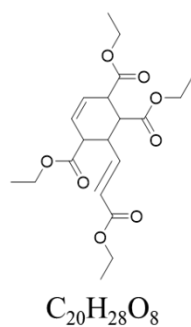
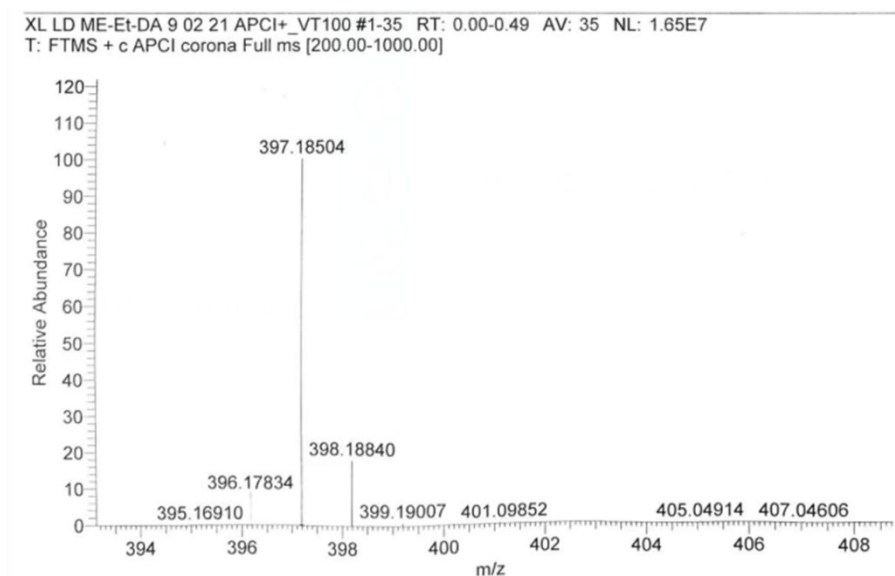


Figure. S17 | Overlays of ^1H NMR spectra of ME-Et depolymerization products in concentrated reaction mixture (100 mg polyME-Et in 1 mL DPE, top) and diluted reaction mixture (100 mg polyME-Et in 10 mL DPE, bottom) (25°C, $CDCl_3$). The three different isomers (EE, EZ and ZZ) are labeled in black, blue and red respectively. The pink arrows indicate the possible D-A product produced, with extra protons on double bonds at 5-7 ppm and two groups of protons on the carbon ring at 2.5-3.5 ppm.



HRMS (APCI) calcd. for $C_{20}H_{29}O_8^+$ ($[M+H]^+$): 397.18569; found 397.18504.

Figure. S18 | Mass spectrum of the dimer byproduct.

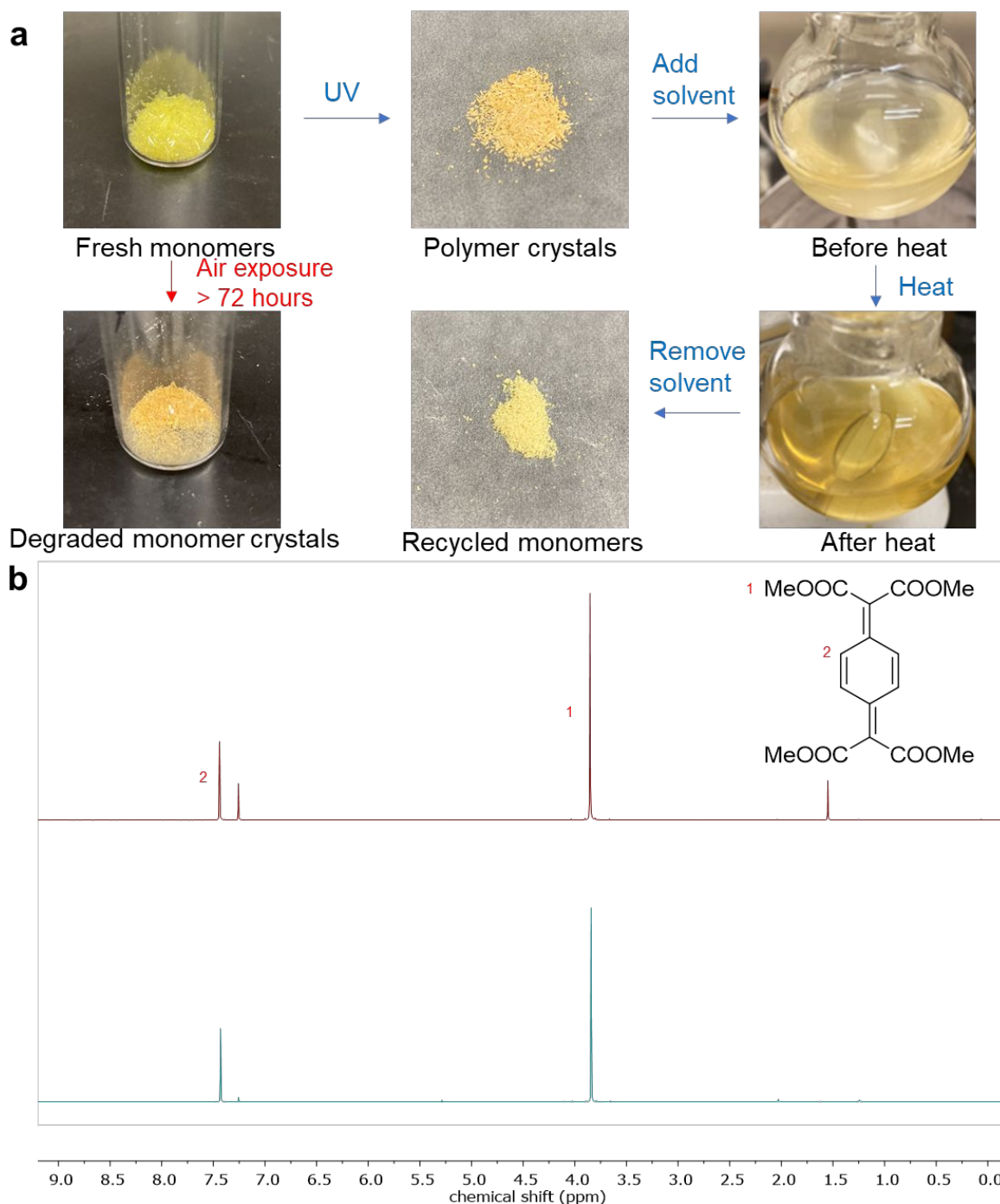


Figure. S19 | a. Photographs of the fresh QDM-Me monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. The bottom left picture shows a partially decomposed monomer after storage in air. The decomposition may be attributed to oxygen interaction and/or visible light induced photopolymerization⁴⁸. **b.** Overlays of ¹H NMR spectra of QDM-Me fresh monomer (top) and recycled monomer via solution-processed method (bottom) (25°C, CDCl₃).

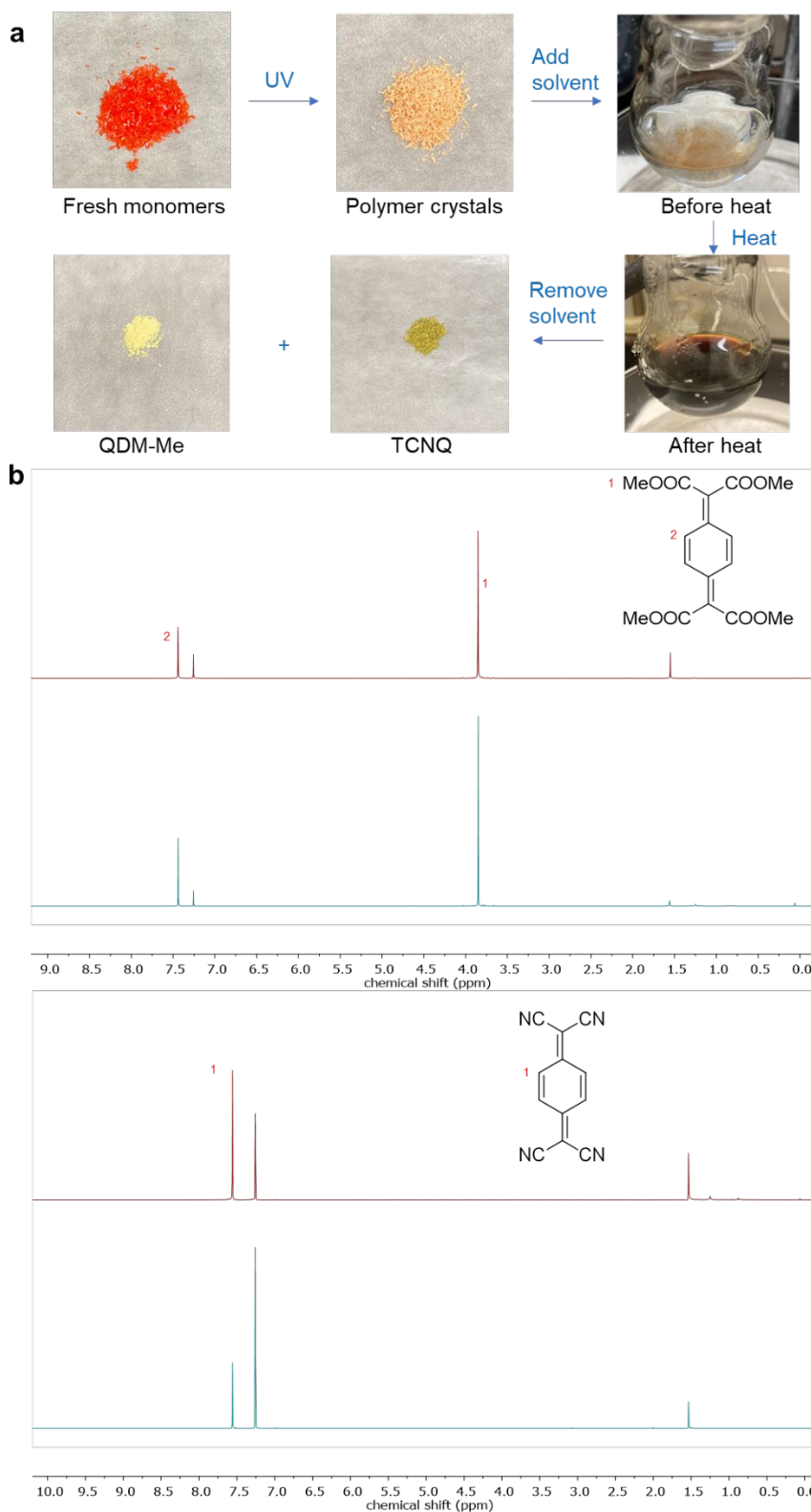


Figure. S20 | a. Photographs of the fresh QDM-Me monomer crystals, polymer crystals, and recycled monomers showing the complete monomer-to-monomer cycle. The depolymerization provides the two monomers QDM-Me and TCNQ. **b.** Overlays of ^1H NMR spectra of QDM-Me/TCNQ fresh monomer (top) and recycled monomer via solution-processed method (bottom) (25°C, CDCl_3).

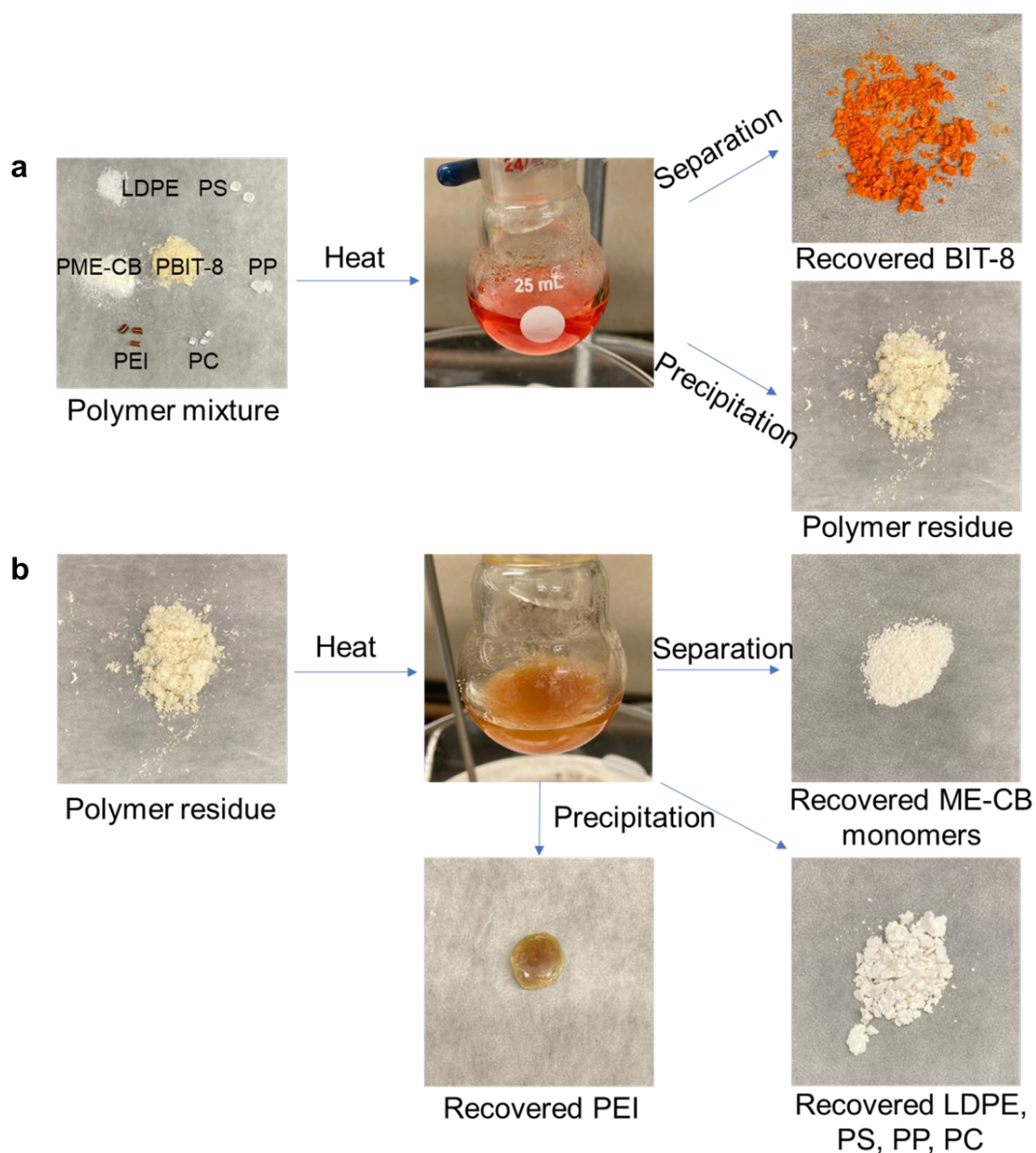


Figure. S21 | **a.** Demonstration of the separation process of polyBIT-8 from polyME-CB, low-density polyethylene (LDPE), polystyrene (PS), polypropylene (PP), polycarbonate (PC) and polyetherimide (PEI). polyBIT-8 was depolymerized and dissolved at 155 °C. All other polymers were precipitated in hexanes after cooling. **b.** Demonstration of the separation process of polyME-CB from the polymer residue of polyBIT-8 separation. polyME-CB was depolymerized and dissolved at 258 °C. The brown color of solution was caused by decomposition of trace amount of polyBIT-8 or BIT-8. All other polymers were precipitated in hexanes after cooling. Note that PEI melted at high temperature but remained insoluble. After cooling, PEI was solidified at the bottom of the reaction flask.

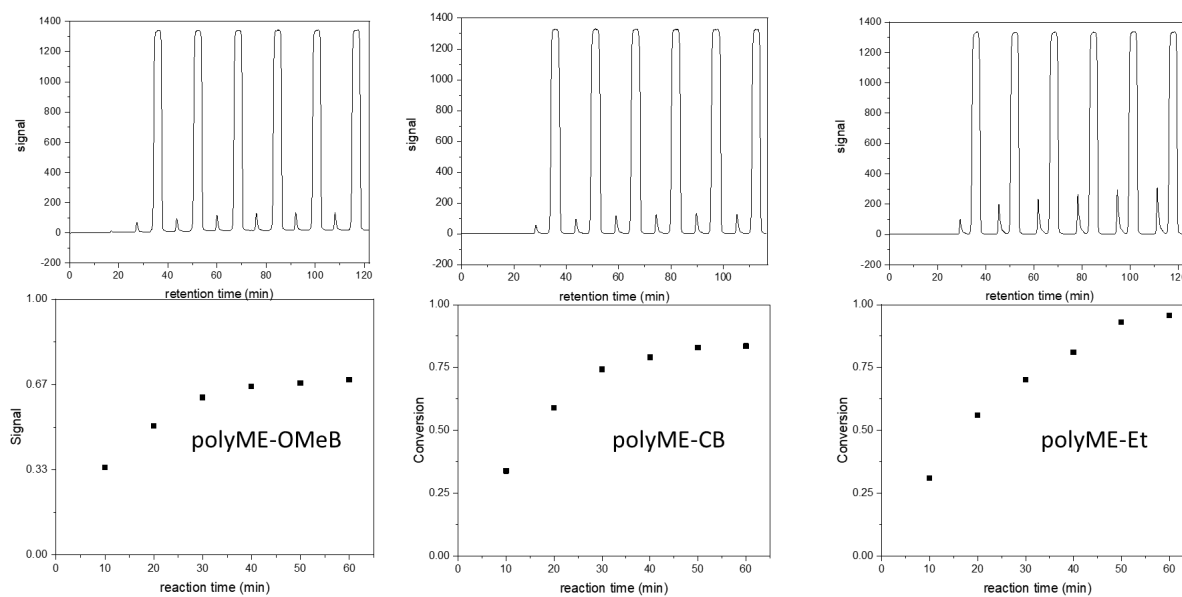


Figure. S22 | HPLC signals at 10 to 60 min reaction time (top) and kinetic plots (bottom) of the representative polyME depolymerizations. The saturated high signals in the HPLC graph belong to solvent DPE.

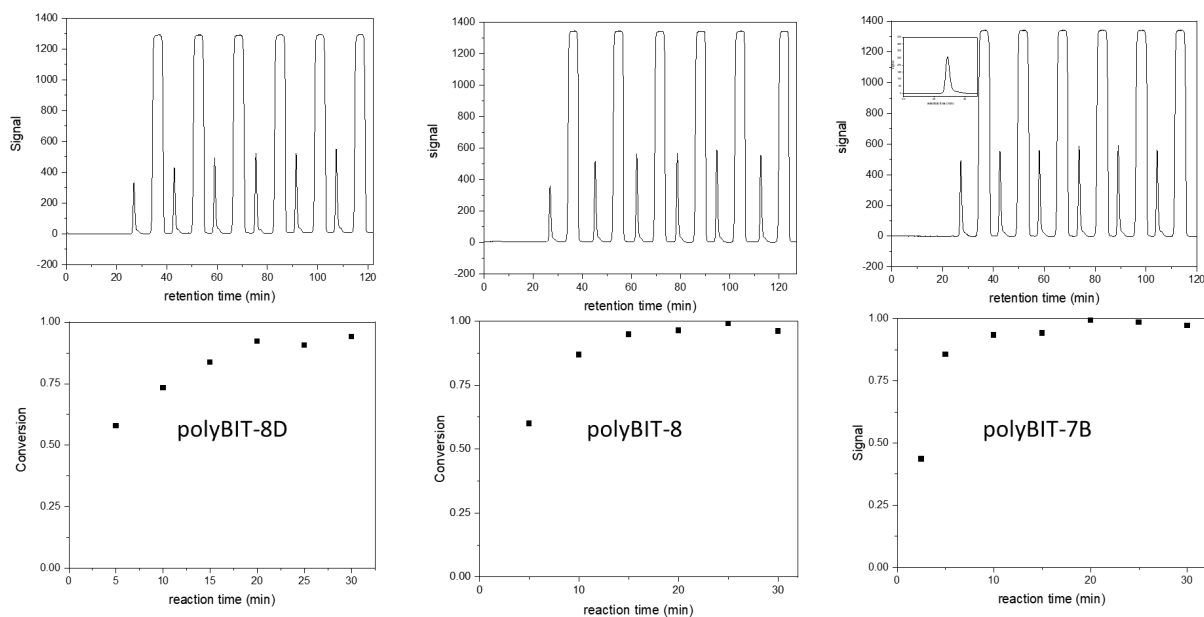


Figure. S23 | HPLC signals at 5 to 30 min reaction time (top) and kinetic plots (bottom) of the representative polyBIT depolymerizations. The inset is the HPLC signal of polyBIT-7B depolymerization mixture after 2.5 min reaction. The saturated high signals in the HPLC graph belong to solvent DPE.

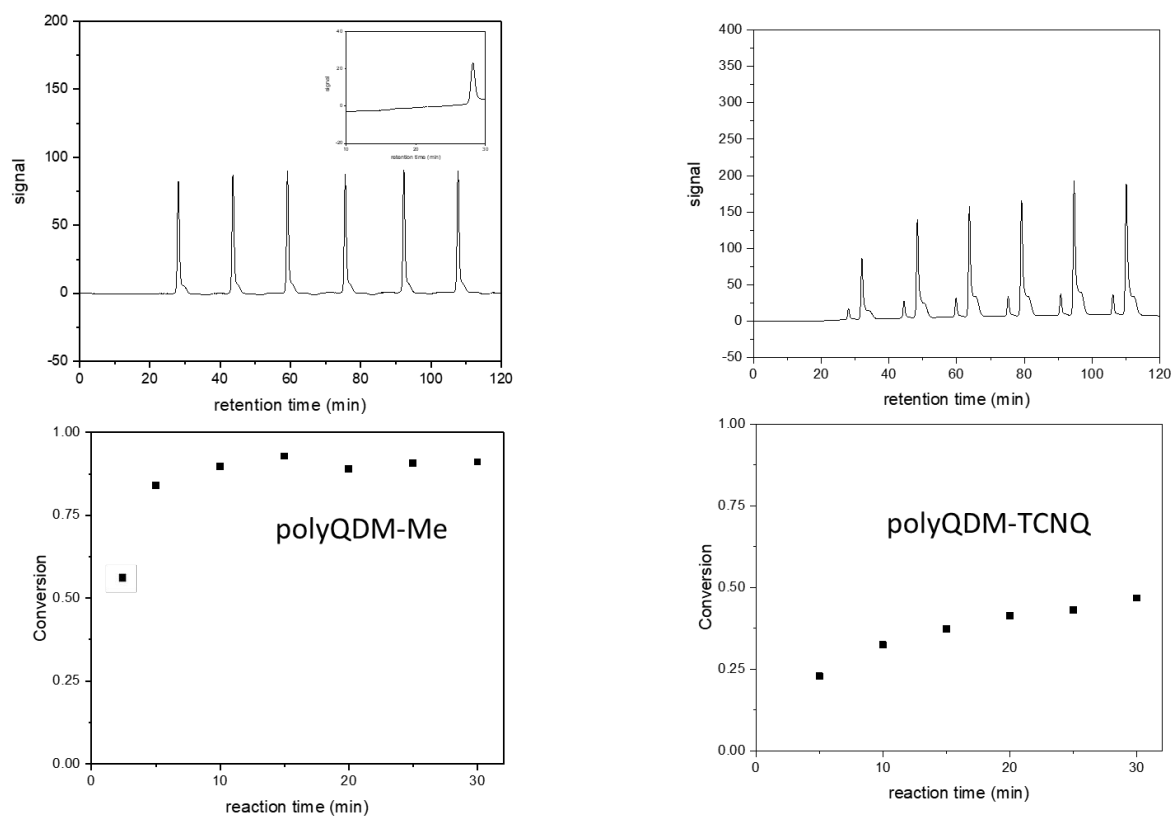


Figure. S24 | HPLC signals at 5 to 30 min reaction time (top) and kinetic plots (bottom) of the representative polyQDM depolymerizations. The inset is the HPLC signal of polyQDM-Me depolymerization mixture after 2.5 min reaction. For polyQDM-TCNQ, the lower peaks belong to TCNQ and higher peaks belong to QDM-Me.

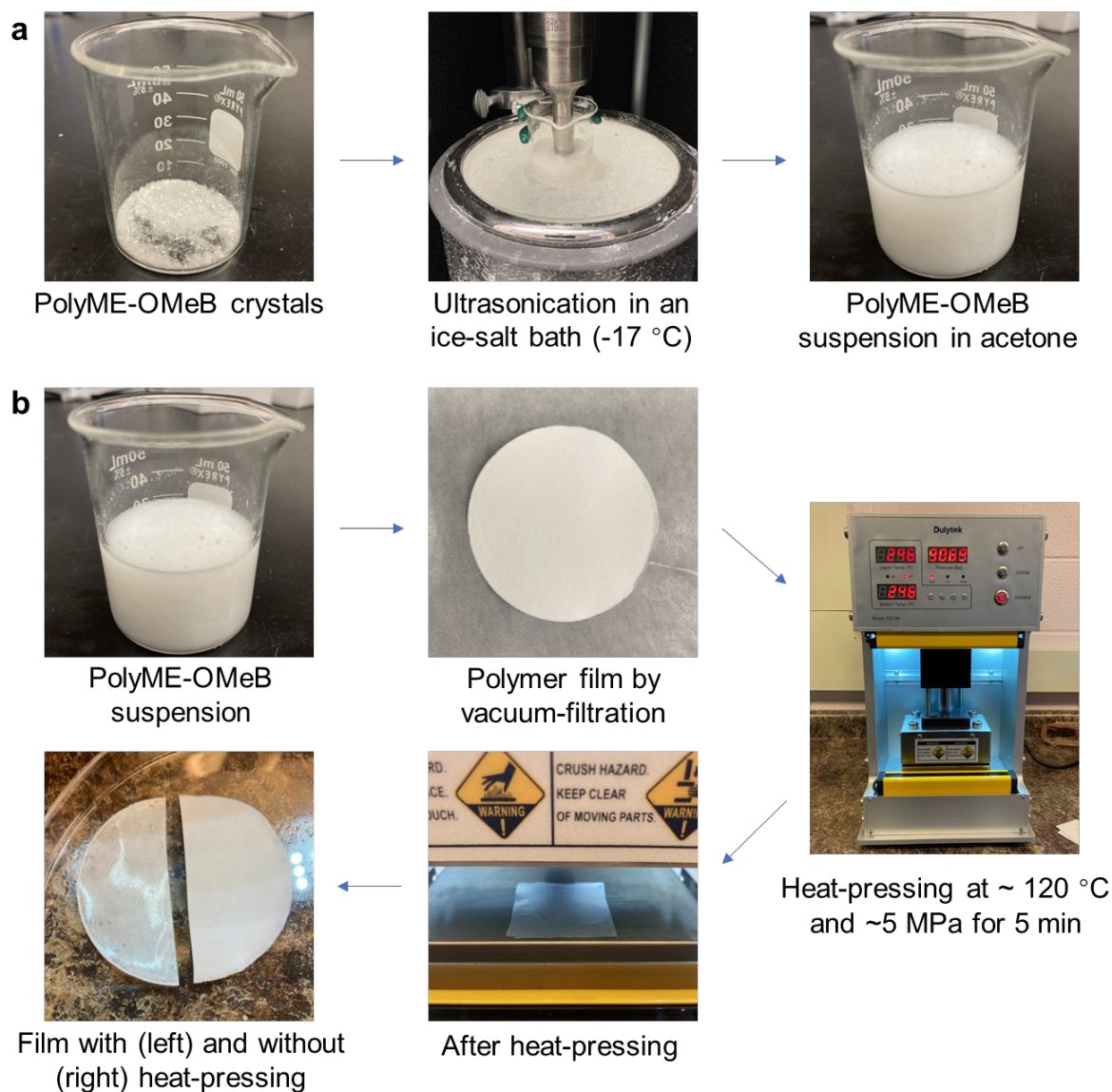


Figure. S25 | a. Photographs of the polyME-OMeB polymer under ultra-sonication and the resulting polymer suspension in acetone. This suspension is meta-stable and maintains without precipitation after 24 h. **b.** Photographs of the polyME-OMeB polymer film fabrication process involving a vacuum-filtration and gentle heat-pressing. Heat-pressed films are semi-transparent and thus look “darker” than untreated white pristine films. The glossy characteristic of the heat-pressed film indicates that its surface is pressed to a smooth finish.

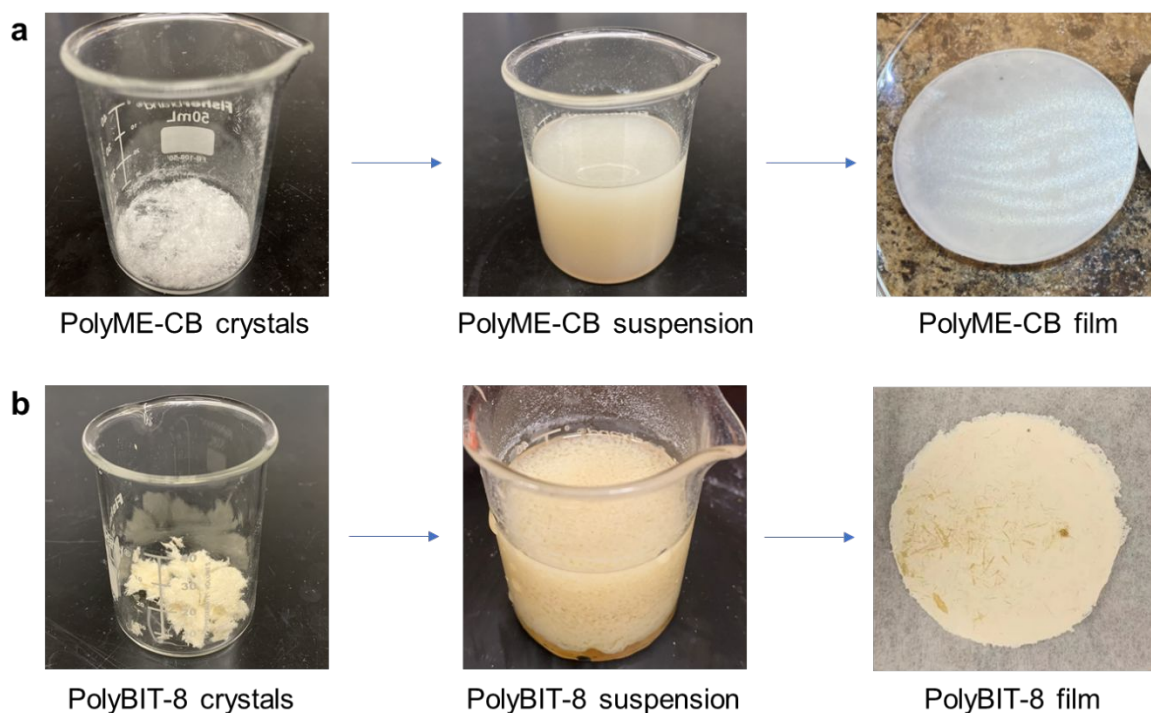


Figure. S26 | a. Photographs of the film fabrication process for polyME-CB. This polymer did not swell in acetone well during the ultra-sonication process and left crude fibers on the film surface. The polymer swelled properly in chloroform and provided with a glossy film after heat-pressing. **b.** Photographs of the film fabrication process for polyBIT-8. Dark patterns are trace amount of BIT-8 monomer generated from mechano-depolymerization.

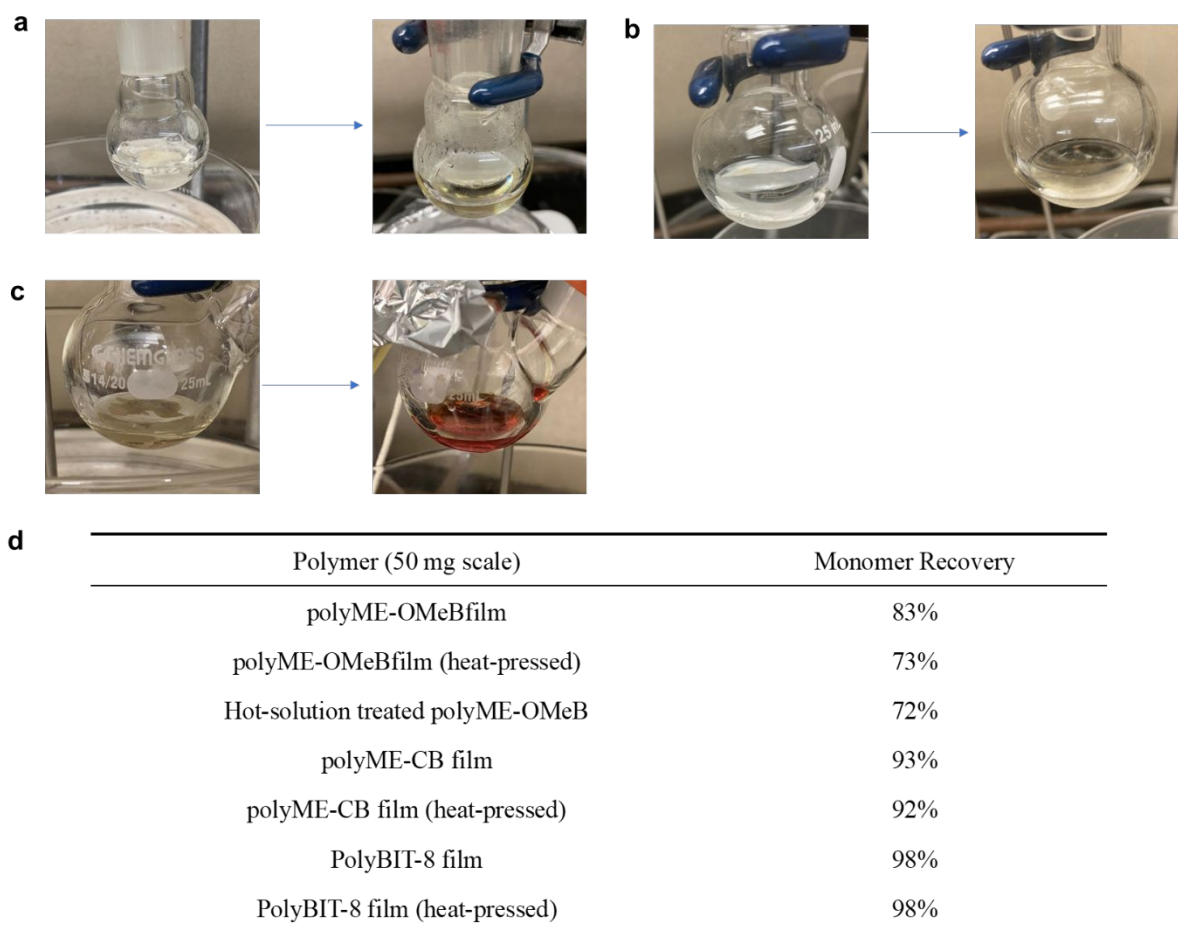


Figure. S27 | Depolymerization reaction of the sonication processed polymer films. **a.** polyME-OMeB, before (left) and after (right) heat at 250 °C; **b.** polyME-CB, before (left) and after (right) heat at 250 °C; **c.** polyBIT-8, before (left) and after (right) heat at 150 °C. **d.** A summary of monomer (monomer isomers for polyME) recovery yields for polymers with different treatments. It can be seen that sonication and mechanical deformation do not influence the recovery yield but heat-treatment indeed leads to a ~10% drop of recovery yield for polyME-OMeB. However, similar experiments were carried out on polyME-CB and polyBIT-8 as well, but no decrease of recovery yields were observed in these polymers (i.e., excellent yields of 92% and 98% were obtained after heat-pressing, respectively).

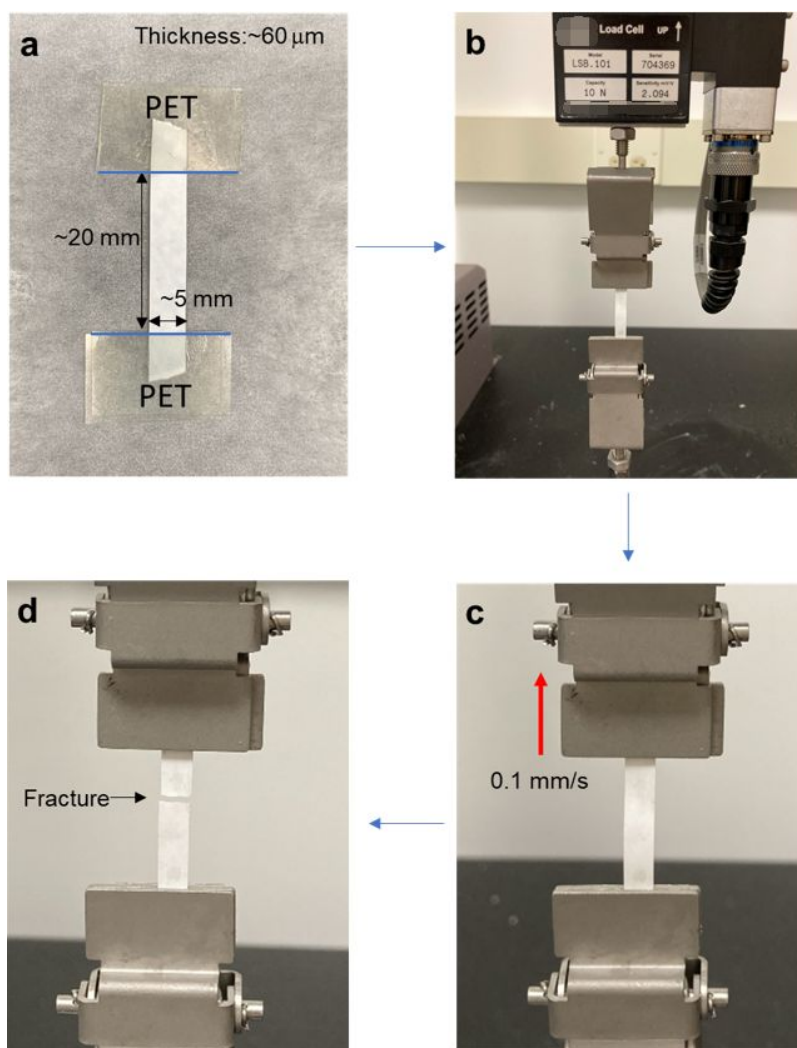


Figure. S28 | Photographs of **a.** a polyME-OMeB sample stripe on PET substrates and its dimension, **b.** the polymer sample fixed vertically on the tensile test machine and the 10 N load cell, **c.** the polymer sample during a tensile test with a crosshead speed of 0.1 mm/s, and **d.** fracture of the polymer sample.

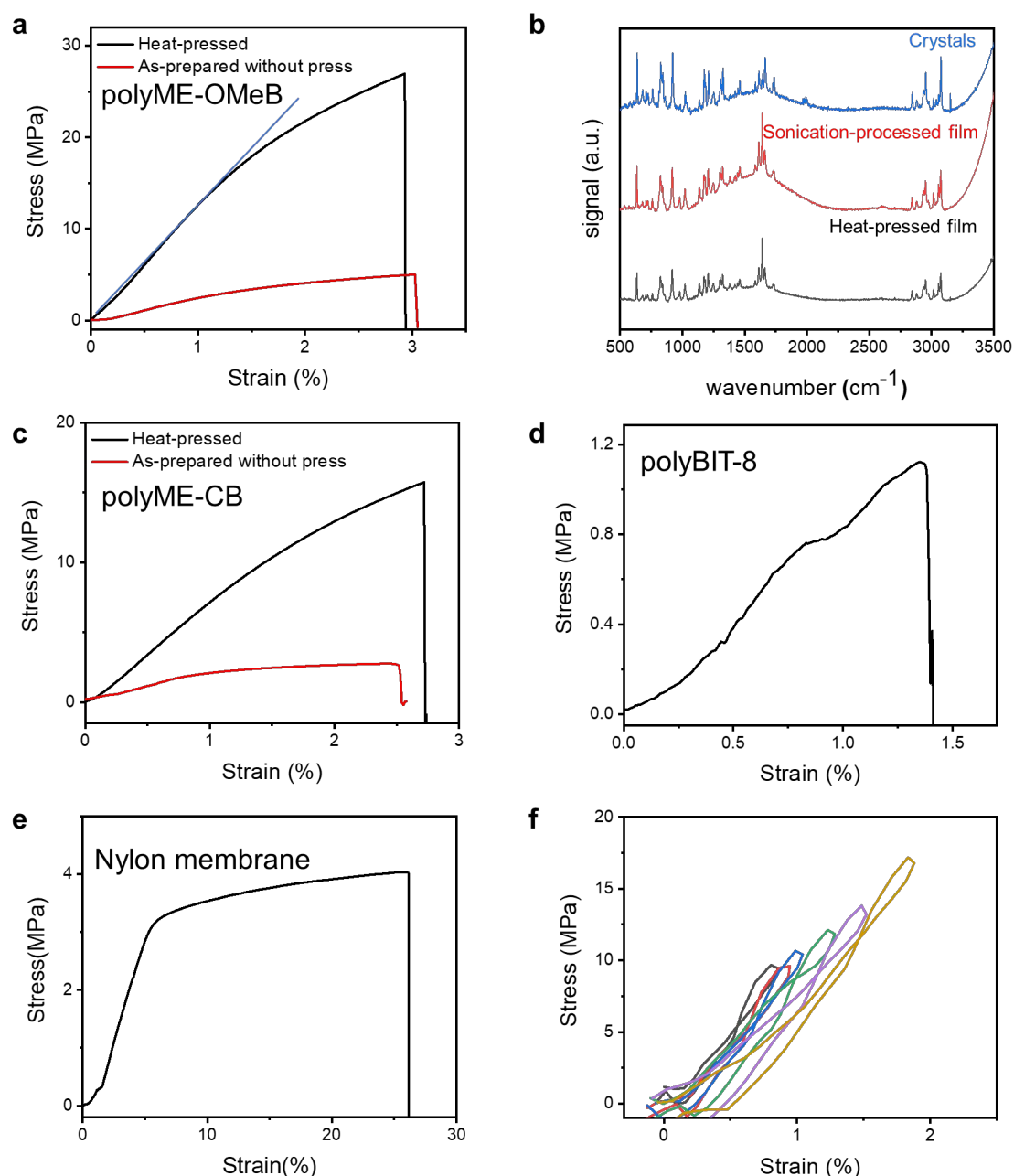


Figure. S29 | **a.** Representative engineering tensile stress-strain curves of sonicated-processed polyME-OMeB films before and after heat pressing. **b.** Raman spectra of polyME-OMeB polymer crystals and sonicated processed films. No significant change was observed from sonication and the heat pressing treatment. **c.** Representative engineering tensile stress-strain curves of polyME-CB films before and after heat pressing (average Young's modulus $E = 0.81$ GPa for heat-pressed film). **d.** A representative engineering tensile stress-strain curve of heat-pressed polyBIT-8 film ($E = 0.12$ GPa). The polymer film before heat pressing was too brittle for tensile stress-strain tests. **e.** A representative engineering tensile stress-strain curve of a commercial nylon filtration membrane (Whatman membrane filters nylon, pore size $0.45\ \mu\text{m}$, $E = 0.11$ GPa). **f.** Cyclic tensile-strain tests for determination of the elastic regime of sonicated-processed polyME-OMeB film.

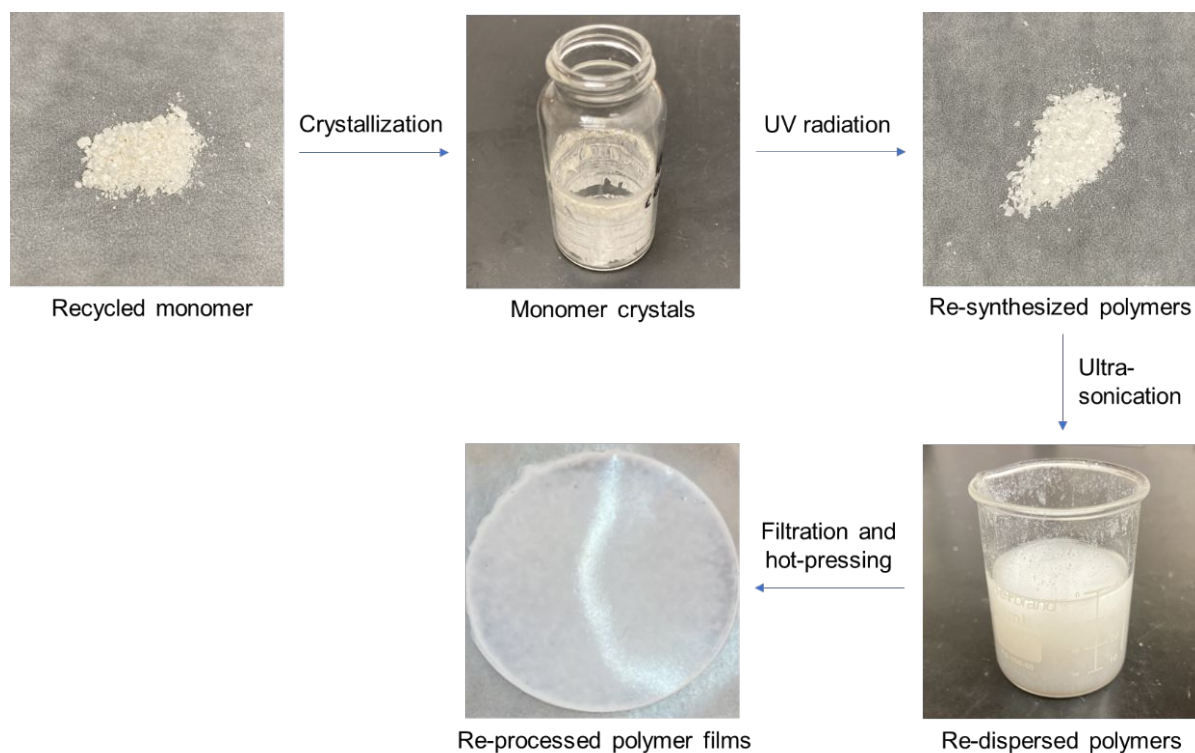
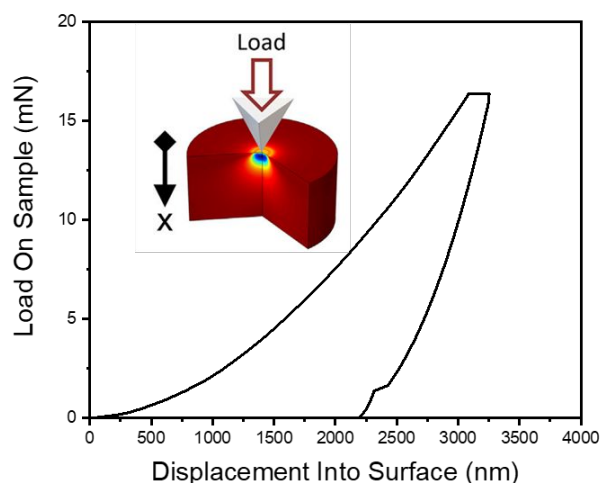


Figure. S30 | Photographs of the recrystallized recycled ME-OMeB monomer (by slow evaporation of DCM solution at room temperature); recycled polyME-OMeB polymer crystals; sonicated recycled polyME-OMeB suspension in acetone; and recycled polyME-OMeB film. Like fresh polymer films, the re-processed polyME-OMeB film is semi-transparent and glossy.



PolyME-OMeB	
Modulus by indentation (GPa)	3.32±0.75
Modulus by tensile test (GPa)	1.18±0.09
Hardness by indentation (MPa)	108±31

Figure. S31 | Left: A typical load versus displacement plot of polyME-OMeB film and schematic of the nanoindentation experiments. Right: A summary of Young's modulus and hardness of polyME-OMeB film measured by nanoindentation tests and tensile stress-strain tests. Note that the porous structure/volume of film under deformation leads to a higher modulus in indentation tests.

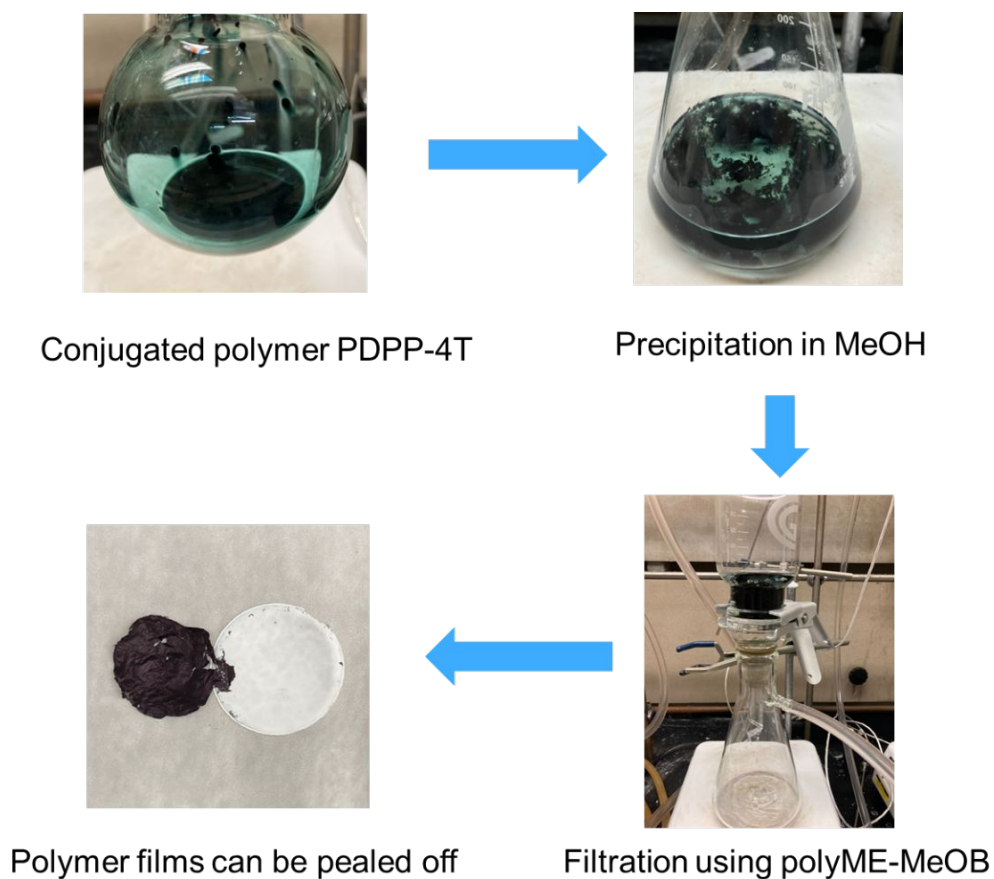


Figure. S32 | Photographs of the conjugated polymer PDPP-4T solution, precipitation of the solution in methanol, filtration of the polymer solvent mixture using polyME-OMeB sonication-processed thin films, and separation of PDPP-4T and polyME-OMeB thin films.

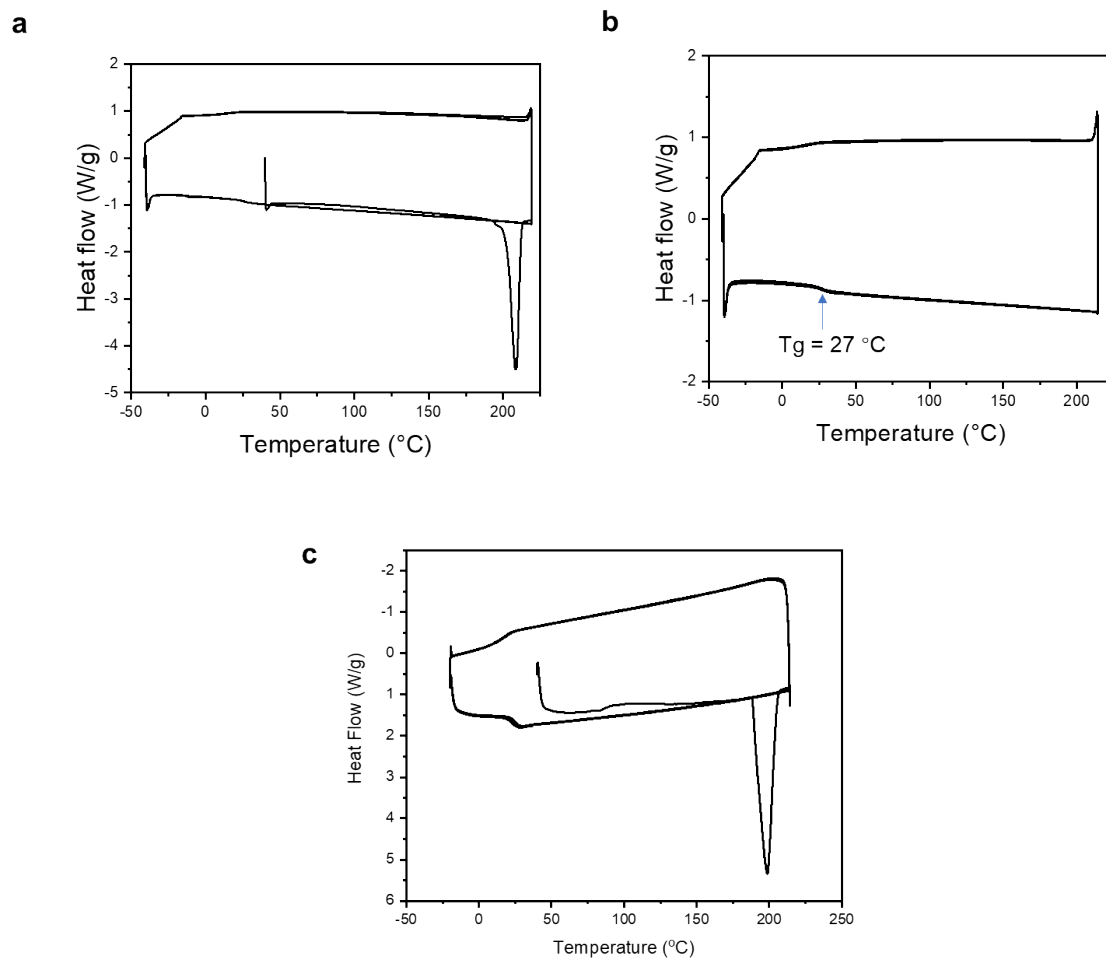


Figure. S33 | Differential scanning calorimetry cyclic plots of **a.** polyME-OMeB crystals, **b.** the solution-processed polyME-OMeB solid, and **c.** the sonication-processed polyME-OMeB polymer film.

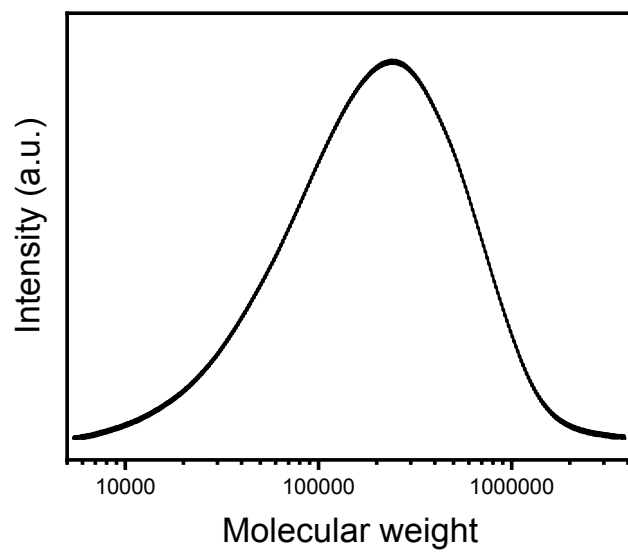


Figure. S34 | Representative size exclusion chromatography elution profile of the solution-processed polyME-OMeB polymer.

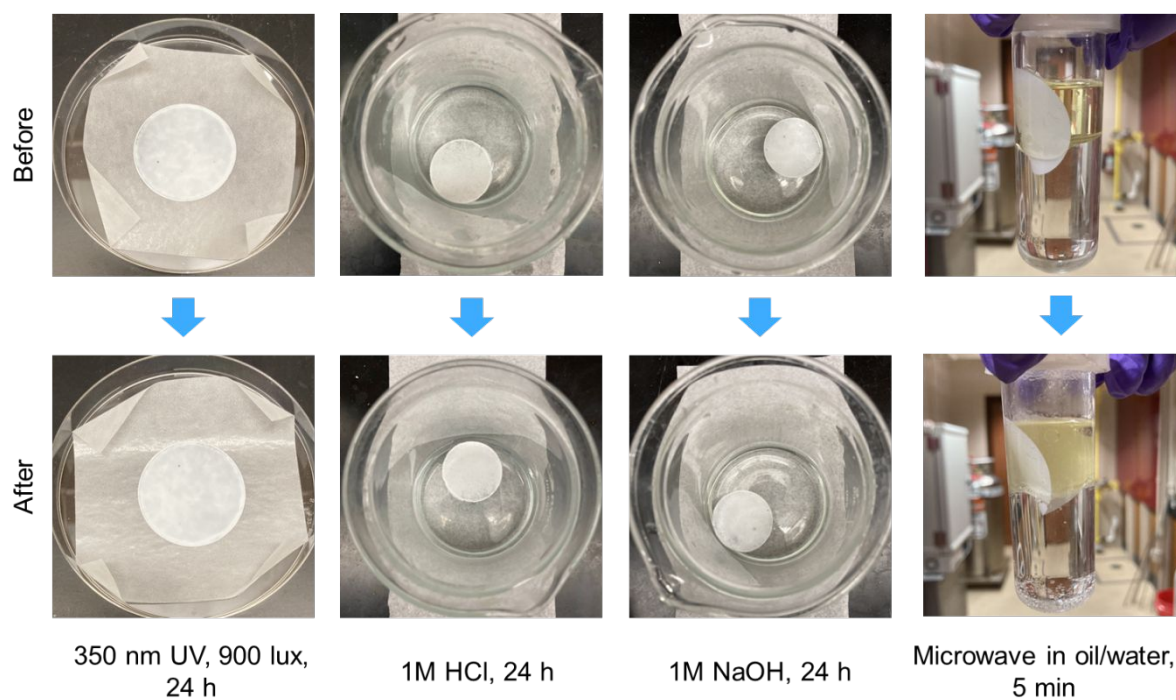


Figure. S35 | Photographs of sonication-processed polyME-OMeB films before and after treatment with strong 350 nm UV (~900 lux), acid (pH~0), base (pH~14) for 24 hours, and microwave heating (95 °C) for 5 min in water/vegetable oil, respectively. All films held their original shapes, appearance, and masses after treatments.

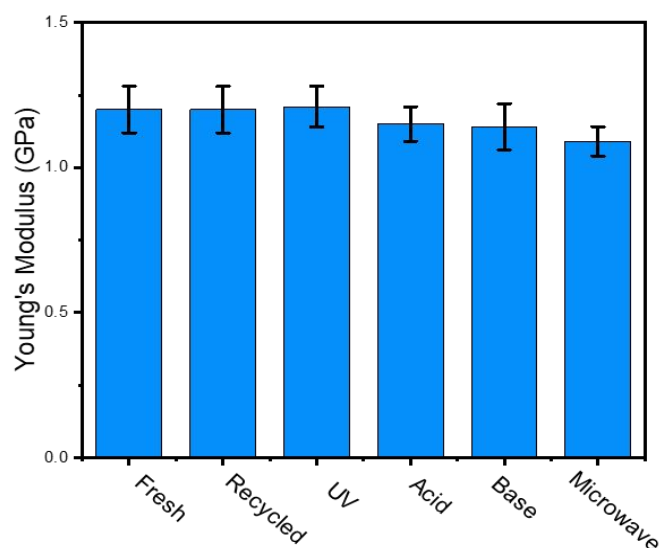


Figure. S36 | Statistics of Young's modulus for the polyME-OMeB films under different treatments. The slight variations between different columns are within experimental error (error bars are labeled on top of each column). We note that, although 5 min microwave treatment did not lead to obvious degradation, a longer time (30 min) microwave treatment do lead to ~20% drop of Young's Modulus, but no chemical degradation was detected.

Table S1 | DFT calculated Bond Dissociation Energies (BDEs) in kcal/mol.

	Bond Type	with Relaxed Dimer	with Crystal
PolyBIT-Me	C-C (between)	43.27	44.07
	C=C (within)	162.35	163.15
PolyME-Me (ZZ)	C-C (between)	51.83	52.19
	C-C (within)	97.79	98.15
	C=C (within)	177.49	177.85
PolyQDM-Me	C-C (between)	59.12	52.98
	C-C (within)	78.14	72.00
Polyethylene	C-C (between)	85.31	n/a
Polystyrene	C-C (between)	76.78	n/a
Polymethylmethacrylate	C-C (between)	72.77	n/a
Ethane	C-C	90.60	n/a
Ethene	C-C	172.95	n/a
Ethyne	C-C	228.76	n/a
Compound 2	C-C	60.94	n/a

Table S2 | Enthalpies, electronic energies, and corrections (au) for small molecules.

	Total Enthalpy	Electronic Energy	Zero Point Energy	Thermal Corrections
Ethane	-79.7553	-79.834	0.0751	0.0044
Radical1,2	-39.8055	-39.8394	0.0299	0.0040
Ethene	-78.5364	-78.5918	0.0514	0.0040
Radical1,2	-39.1304	-39.1515	0.0173	0.0038
Ethyne	-77.3029	-77.3322	0.0257	0.0036
Radical1,2	-38.4692	-38.4790	0.0065	0.0033
Compound2	-1089.4147	-1090.0530	0.6197	0.0185
Radical1	-544.6589	-544.9742	0.3056	0.0096
Radical2	-544.6587	-544.9741	0.3057	0.0096

Table S3 | Enthalpies, electronic energies, and corrections (au) for common polymers.

	Total Enthalpy	Electronic Energy	Zero Point Energy	Thermal Corrections
Polyethylene (PE)				
Dimer	-236.9011	-237.1006	0.1901	0.0093
Radical1	-118.3826	-118.4762	0.0884	0.0052
Radical2	-118.3826	-118.4762	0.0884	0.0052
Polystyrene (PS)				
Dimer	-698.8433	-699.2158	0.3547	0.0178
Radical1	-349.3500	-349.5311	0.1711	0.0100
Radical2	-349.3709	-349.5523	0.1728	0.0086
Polymethylmethacrylate (PMMA)				
Dimer	-771.1694	-771.5239	0.3344	0.0201
Radical1	-385.5169	-385.6891	0.1611	0.0111
Radical2	-385.5365	-385.7097	0.1629	0.0103

Table S4 | Enthalpies, electronic energies, and corrections (au) for topochemical polymers.

	Total Enthalpy	Electronic Energy	Zero Point Energy	Thermal Corrections
PolyBIT-Me				
Crystal	-2675.6234	-2676.3770	0.7026	0.0509
Dimer	-2675.6222	-2676.3800	0.7059	0.0519
Bond1 Radical1,2 ^a	-1337.7766	-1338.1527	0.3498	0.0263
Bond2 Radical1 ^b	-1986.6473	-1987.1924	0.5068	0.0383
Bond2 Radical2 ^b	-688.7161	-688.9221	0.1920	0.0140
PolyME-Me (ZZ)				
Crystal	-1303.0777	-1303.5407	0.4319	0.0310
Dimer	-1303.0771	-1303.5381	0.4316	0.0294
Bond1 Radical1,2 ^a	-651.4973	-651.7252	0.2120	0.0160
Bond2 Radical1 ^c	-918.6223	-918.9275	0.2830	0.0222
Bond2 Radical2 ^c	-384.2989	-384.4482	0.1388	0.0105
Bond3 Radical1 ^b	-957.2232	-957.5428	0.2978	0.0217
Bond3 Radical2 ^b	-345.5711	-345.7014	0.1207	0.0096
PolyQDM-Me				
Crystal	-2521.6702	-2522.4170	0.6935	0.0533
Dimer	-2521.6800	-2522.4296	0.6970	0.0526
Bond1 Radical1,2 ^a	-1260.7929	-1261.1662	0.3459	0.0275
Bond2 radical1 ^c	-1755.7921	-1756.2903	0.4605	0.0377
Bond2 radical2 ^c	-765.7634	-766.0116	0.2313	0.0169

^a C-C between monomers

^b C=C within monomers

^c C-C within monomers

Table S5 | Statistics of the tensile stress-strain tests of polyME-OMeB films.

Treatment	Young's Modulus (GPa)	Ultimate strength (MPa)	Strain at break (%)
Pristine	1.20±0.08	23±4	2.5±0.5
#1	1.13	17	1.9
#3	1.27	23	2.1
#4	1.13	26	3.1
#5	1.32	28	3.0
#6	1.17	20	2.2
UVA	1.21±0.07	24±3	2.6±0.5
#1	1.11	19	1.9
#2	1.15	26	2.8
#3	1.31	28	3.5
#4	1.27	23	2.3
#5	1.21	23	2.4
Base	1.14±0.08	21±1	2.3±0.3
#1	1.03	22	2.7
#2	1.24	20	1.9
#3	1.15	20	2.2
Acid	1.15±0.06	19±2	2.0±0.3
#1	1.20	22	2.2
#2	1.15	17	1.5
#3	1.20	19	1.9
#4	1.06	16	2.2
Microwave	1.09±0.05	22±1	2.9±0.4
#1	1.10	23	3.2
#2	1.14	20	2.3
#3	1.00	22	3.3
#4	1.12	22	2.6

Recycled	1.20±0.08	20±2	2.7±0.4
#1	1.26	20	2.3
#3	1.15	19	2.3
#4	1.31	24	3.0
#5	1.10	18	3.3
Extruded Filament	0.49±0.02	20±0.5	199±8
#1	0.51	20	205
#2	0.47	20	188
#3	0.48	19	204

The stress σ and strain ε were calculated using equations shown below:

$$\sigma = \frac{F}{A_0} = \frac{F}{H \times W}, \quad \varepsilon = \frac{L - L_0}{L_0}$$

The axial length L_0 and width W were measured by a digital caliper, and the thickness H was measured by a micro screw gauge.

Young's modulus was calculated by slope of the linear interval of the stress-strain curve, i.e. σ/ε . Ultimate strength and Strain at break are the stress and strain at the point of failure, respectively.

Specifically, for hot-solvent treated polymer filaments, the cross-sectional area A_0 of the testing samples were calculated from the radius of filaments. Ultimate strength and strain are the stress and strain at the point of yield, respectively.

II. Appendix (single crystal structure data)

polyBIT-6 (see CCDC-2088147 for more complete data)

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

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;
```

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'x, -y-1/2, z-1/2'

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SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

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Reflections were merged by SHELXL according to the crystal class for the calculation of statistics and refinement.

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SHELXLE Rev1149 (Hübschle et al., 2011)
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C12 0.0357(10) 0.0284(12) 0.0255(11) -0.0025(8) 0.0105(9) 0.0013(8)
C13 0.0371(10) 0.0342(13) 0.0287(12) -0.0033(9) 0.0081(9) 0.0002(9)
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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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 C14 C15 H15A 109.5 . . ?
 C14 C15 H15B 109.5 . . ?
 H15A C15 H15B 109.5 . . ?
 C14 C15 H15C 109.5 . . ?
 H15A C15 H15C 109.5 . . ?
 H15B C15 H15C 109.5 . . ?

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
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
 C1 C1 C2 O1 26.2(4) 3_556 . . . ?
 C9 C1 C2 O1 -155.0(2) ?
 C1 C1 C2 C3 -155.2(2) 3_556 . . . ?
 C9 C1 C2 C3 23.62(18) ?
 O1 C2 C3 C8 167.95(19) ?
 C1 C2 C3 C8 -10.7(2) ?
 O1 C2 C3 C4 -13.8(3) ?
 C1 C2 C3 C4 167.55(19) ?
 C8 C3 C4 C5 2.5(3) ?
 C2 C3 C4 C5 -175.61(19) ?
 C3 C4 C5 C6 0.8(3) ?
 C4 C5 C6 C7 -2.8(3) ?
 C5 C6 C7 C8 1.3(3) ?
 C4 C3 C8 C7 -3.9(3) ?
 C2 C3 C8 C7 174.47(17) ?

C4 C3 C8 C9 174.17(17) ?
 C2 C3 C8 C9 -7.4(2) ?
 C6 C7 C8 C3 2.0(3) ?
 C6 C7 C8 C9 -175.81(18) ?
 C10 O2 C9 C8 -65.0(2) ?
 C10 O2 C9 C1 55.2(2) ?
 C10 O2 C9 C9 176.44(17) . . . 3_566 ?
 C3 C8 C9 O2 152.49(16) ?
 C7 C8 C9 O2 -29.6(3) ?
 C3 C8 C9 C1 21.87(19) ?
 C7 C8 C9 C1 -160.19(19) ?
 C3 C8 C9 C9 -95.2(2) . . . 3_566 ?
 C7 C8 C9 C9 82.7(3) . . . 3_566 ?
 C1 C1 C9 O2 27.0(3) 3_556 . . . ?
 C2 C1 C9 O2 -151.67(16) ?
 C1 C1 C9 C8 152.0(3) 3_556 . . . ?
 C2 C1 C9 C8 -26.75(17) ?
 C1 C1 C9 C9 -89.9(3) 3_556 . . 3_566 ?
 C2 C1 C9 C9 91.4(2) . . . 3_566 ?
 C9 O2 C10 O3 0.6(3) ?
 C9 O2 C10 C11 178.87(16) ?
 O3 C10 C11 C12 -20.1(3) ?
 O2 C10 C11 C12 161.69(17) ?
 C10 C11 C12 C13 179.02(18) ?
 C11 C12 C13 C14 177.96(19) ?
 C12 C13 C14 C15 178.2(2) ?

_refine_diff_density_max 0.194
 _refine_diff_density_min -0.321
 _refine_diff_density_rms 0.061

_shelx_res_file

;

TITL PBIT_Hex2_0m in P2(1)/c

PBIT_Hex2_0m.res

created by SHELXL-2018/3 at 13:10:48 on 17-Oct-2020

CELL 1.54178 13.67610 4.86760 19.51350 90.0000 110.2522 90.0000

ZERR 4.00 0.00050 0.00020 0.00060 0.0000 0.0021 0.0000

LATT 1

SYMM -X, 0.5+Y, 0.5-Z

SFAC C H O

UNIT 60 60 12

LIST 6 ! automatically inserted. Change 6 to 4 for CHECKCIF!!

TEMP -123.150

SIZE 0.55 0.09 0.05

L.S. 4

BOND \$H

ACTA

CONF

FMAP 2

PLAN 20

WGHT 0.073300 0.236700

FVAR 0.71236

O1 3 -0.065873 -0.046841 0.351390 11.00000 0.04559 0.02348 =
0.02007 -0.00319 0.01003 -0.00645

O2 3 0.114635 0.456141 0.576915 11.00000 0.03029 0.01916 =
0.01851 -0.00136 0.00792 -0.00073

O3 3 0.235688 0.141986 0.576445 11.00000 0.03923 0.03293 =
0.02821 -0.00803 0.00946 0.00615

C1 1 0.008127 0.104972 0.479980 11.00000 0.02844 0.01914 =
0.01961 -0.00063 0.01100 0.00169

C2 1 -0.007676 0.098681 0.398353 11.00000 0.03443 0.01849 =
0.01915 0.00142 0.01079 0.00259

C3 1 0.060954 0.312855 0.386625 11.00000 0.03430 0.01846 =
0.02001 0.00211 0.01108 0.00178

C4 1 0.089371 0.358443 0.325697 11.00000 0.04178 0.02190 =
0.02236 0.00008 0.01385 0.00086

AFIX 43

H4 2 0.060451 0.252371 0.282509 11.00000 -1.20000

AFIX 0

C5 1 0.161464 0.564375 0.330253 11.00000 0.04235 0.02638 =
0.02894 0.00429 0.02036 -0.00036

AFIX 43

H5 2 0.181778 0.602148 0.289323 11.00000 -1.20000

AFIX 0

C6 1 0.204206 0.715680 0.393833 11.00000 0.03640 0.02485 =
0.03175 0.00039 0.01773 -0.00290

AFIX 43

H6 2 0.255700 0.850030 0.396297 11.00000 -1.20000

AFIX 0

C7 1 0.173640 0.675968 0.454286 11.00000 0.03266 0.02146 =
0.02597 -0.00172 0.01348 -0.00031

AFIX 43

H7 2 0.202130 0.782914 0.497358 11.00000 -1.20000

AFIX 0

C8 1 0.099701 0.473221 0.448849 11.00000 0.03229 0.01652 =

			0.02055	0.00214	0.01279	0.00156		
C9	1		0.048309	0.400118	0.503709	11.00000	0.03213	0.01758 =
			0.01587	-0.00003	0.00828	-0.00108		
C10	1		0.204920	0.317153	0.607784	11.00000	0.03159	0.02278 =
			0.02396	-0.00068	0.01058	0.00027		
C11	1		0.259686	0.419716	0.683577	11.00000	0.03518	0.02544 =
			0.02516	-0.00410	0.00718	0.00029		
AFIX	23							
H11A	2		0.207552	0.453079	0.707393	11.00000	-1.20000	
H11B	2		0.293378	0.597499	0.680959	11.00000	-1.20000	
AFIX	0							
C12	1		0.341687	0.222391	0.730335	11.00000	0.03568	0.02842 =
			0.02551	-0.00251	0.01048	0.00133		
AFIX	23							
H12A	2		0.393394	0.186516	0.706301	11.00000	-1.20000	
H12B	2		0.307976	0.045603	0.733898	11.00000	-1.20000	
AFIX	0							
C13	1		0.397847	0.332947	0.806943	11.00000	0.03707	0.03417 =
			0.02872	-0.00325	0.00814	0.00016		
AFIX	23							
H13A	2		0.433172	0.506768	0.803085	11.00000	-1.20000	
H13B	2		0.345468	0.375833	0.829920	11.00000	-1.20000	
AFIX	0							
C14	1		0.477430	0.138364	0.856019	11.00000	0.04678	0.05622 =
			0.03192	0.00040	0.00629	0.01337		
AFIX	23							
H14A	2		0.528509	0.090831	0.832281	11.00000	-1.20000	
H14B	2		0.441685	-0.033234	0.861106	11.00000	-1.20000	
AFIX	0							
C15	1		0.535696	0.251097	0.931440	11.00000	0.05621	0.07082 =
			0.03688	0.00345	-0.00210	-0.00664		
AFIX	137							
H15A	2		0.573220	0.418087	0.927189	11.00000	-1.50000	
H15B	2		0.585534	0.113512	0.959977	11.00000	-1.50000	
H15C	2		0.486068	0.294695	0.955993	11.00000	-1.50000	
AFIX	0							
HKLF	4							

REM PBIT_Hex2_0m in P2(1)/c

REM wR2 = 0.1480, GooF = S = 1.056, Restrained GooF = 1.056 for all data

REM R1 = 0.0508 for 1801 Fo > 4sig(Fo) and 0.0749 for all 2544 data

REM 164 parameters refined using 0 restraints

END

WGHT 0.0731 0.2358

REM Highest difference peak 0.194, deepest hole -0.321, 1-sigma level 0.061

Q1	1	-0.0159	0.5003	0.3869	11.00000	0.05	0.19
Q2	1	0.2031	-0.1004	0.5984	11.00000	0.05	0.19
Q3	1	0.3156	-0.2064	0.7637	11.00000	0.05	0.19
Q4	1	0.3895	0.6198	0.8431	11.00000	0.05	0.19
Q5	1	0.1699	0.4839	0.4734	11.00000	0.05	0.18
Q6	1	0.1572	0.1049	0.5273	11.00000	0.05	0.18
Q7	1	0.1136	0.1110	0.3551	11.00000	0.05	0.18
Q8	1	0.1139	0.6337	0.4281	11.00000	0.05	0.18
Q9	1	0.5192	-0.1230	0.8571	11.00000	0.05	0.18
Q10	1	0.1929	0.7116	0.6594	11.00000	0.05	0.17
Q11	1	0.1060	0.3510	0.4182	11.00000	0.05	0.17
Q12	1	0.1919	0.2582	0.3591	11.00000	0.05	0.17
Q13	1	0.2143	0.7063	0.6085	11.00000	0.05	0.17
Q14	1	0.1521	0.7905	0.3413	11.00000	0.05	0.17
Q15	1	-0.0407	-0.3199	0.3587	11.00000	0.05	0.17
Q16	1	0.5547	0.5154	0.9376	11.00000	0.05	0.17
Q17	1	0.2388	0.9434	0.3797	11.00000	0.05	0.17
Q18	1	0.4745	-0.1027	0.8168	11.00000	0.05	0.17
Q19	1	0.0921	0.5224	0.3119	11.00000	0.05	0.16
Q20	1	0.1382	0.9691	0.3633	11.00000	0.05	0.16

;

_shelx_res_checksum 84195

polyBIT-7 (see CCDC-2133259 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C16 H17 O3'
_chemical_formula_weight    257.29
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0033 0.0016
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0106 0.0060
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

```
_space_group_crystal_system monoclinic
_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, y+1/2, -z+1/2'
  '-x, -y, -z'
  'x, -y-1/2, z-1/2'
```

_cell_length_a	14.313(5)
_cell_length_b	4.8632(17)
_cell_length_c	19.271(7)
_cell_angle_alpha	90
_cell_angle_beta	104.816(9)
_cell_angle_gamma	90
_cell_volume	1296.8(8)
_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflms_used	9947
_cell_measurement_theta_min	2.3025
_cell_measurement_theta_max	29.6121

_exptl_crystal_description	plate
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.318
_exptl_crystal_F_000	548
_exptl_transmission_factor_min	?
_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.550
_exptl_crystal_size_mid	0.180
_exptl_crystal_size_min	0.150
_exptl_absorpt_coefficient_mu	0.090
_shelx_estimated_absorpt_T_min	0.952
_shelx_estimated_absorpt_T_max	0.987
_exptl_absorpt_correction_type	multi-scan
_exptl_absorpt_correction_T_min	0.6140
_exptl_absorpt_correction_T_max	0.7459
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK α
_diffn_source	'fine focus sealed tube X-ray source'
_diffn_source_current	'30 mA'
_diffn_source_voltage	'50 kV'
_diffn_radiation_monochromator	'Triumph curved graphite crystal'

```

_diffrn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel
array detector (CPAD)
;
_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number           32739
_diffrn_reflns_av_unetI/netI    0.0399
_diffrn_reflns_av_R_equivalents 0.0777
_diffrn_reflns_limit_h_min      -19
_diffrn_reflns_limit_h_max      19
_diffrn_reflns_limit_k_min      -6
_diffrn_reflns_limit_k_max      6
_diffrn_reflns_limit_l_min      -26
_diffrn_reflns_limit_l_max      26
_diffrn_reflns_theta_min        2.186
_diffrn_reflns_theta_max        29.652
_diffrn_reflns_theta_full       25.242
_diffrn_measured_fraction_theta_max 0.997
_diffrn_measured_fraction_theta_full 0.997
_diffrn_reflns_Laue_measured_fraction_max 0.997
_diffrn_reflns_Laue_measured_fraction_full 0.997
_diffrn_reflns_point_group_measured_fraction_max 0.997
_diffrn_reflns_point_group_measured_fraction_full 0.997
_reflns_number_total            3652
_reflns_number_gt               3000
_reflns_threshold_expression     'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

```

```

_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.

```

;

```

```

_computing_data_collection      'Apex4 v2021.4-0 (Bruker, 2021)'

```

```

_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction      'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution  'Isomorphous replacement'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
;
_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details
;
Solved by isomorphous replacement from its not polymerized starting
material.
;
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type          full
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
'w=1/[s^2*(Fo^2)+(0.0657P)^2+1.6801P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary    isomor
_atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens  geom
_refine_ls_hydrogen_treatment   constr
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       .
_refine_ls_number_reflns        3652
_refine_ls_number_parameters     173
_refine_ls_number_restraints     0
_refine_ls_R_factor_all         0.0714
_refine_ls_R_factor_gt          0.0591
_refine_ls_wR_factor_ref        0.1693
_refine_ls_wR_factor_gt         0.1614
_refine_ls_goodness_of_fit_ref   1.087
_refine_ls_restrained_S_all      1.087
_refine_ls_shift/su_max          0.000
_refine_ls_shift/su_mean         0.000

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

O1 O 1.05758(11) 1.0485(3) 0.64582(7) 0.0225(3) Uani 1 1 d

O2 O 0.89475(9) 0.5408(3) 0.42894(6) 0.0155(3) Uani 1 1 d

O3 O 0.78076(10) 0.8549(3) 0.43482(8) 0.0262(3) Uani 1 1 d

C1 C 0.99184(12) 0.8952(3) 0.52030(8) 0.0141(3) Uani 1 1 d

C2 C 1.00451(13) 0.9015(3) 0.60180(9) 0.0158(3) Uani 1 1 d

C3 C 0.94052(13) 0.6869(3) 0.61743(9) 0.0160(3) Uani 1 1 d

C4 C 0.91336(14) 0.6416(4) 0.68097(9) 0.0206(4) Uani 1 1 d

H4 H 0.939869 0.747494 0.722811 0.025 Uiso 1 1 calc R U . . .

C5 C 0.84602(15) 0.4358(4) 0.68076(10) 0.0232(4) Uani 1 1 d

H5 H 0.826246 0.398088 0.723248 0.028 Uiso 1 1 calc R U . . .

C6 C 0.80701(14) 0.2832(4) 0.61839(10) 0.0222(4) Uani 1 1 d

H6 H 0.758770 0.149402 0.618741 0.027 Uiso 1 1 calc R U . . .

C7 C 0.83716(13) 0.3228(4) 0.55598(9) 0.0185(3) Uani 1 1 d

H7 H 0.811718 0.215523 0.514227 0.022 Uiso 1 1 calc R U . . .

C8 C 0.90587(12) 0.5253(3) 0.55714(9) 0.0151(3) Uani 1 1 d

C9 C 0.95485(12) 0.5994(3) 0.49863(8) 0.0138(3) Uani 1 1 d

C10 C 0.81070(13) 0.6786(4) 0.40275(9) 0.0174(3) Uani 1 1 d

C11 C 0.76324(14) 0.5694(4) 0.32914(10) 0.0209(4) Uani 1 1 d

H11A H 0.746599 0.373643 0.333440 0.025 Uiso 1 1 calc R U . . .

H11B H 0.810259 0.578039 0.299414 0.025 Uiso 1 1 calc R U . . .

C12 C 0.67226(14) 0.7237(4) 0.29067(10) 0.0225(4) Uani 1 1 d

H12A H 0.621719 0.695990 0.316751 0.027 Uiso 1 1 calc R U . . .

H12B H 0.686534 0.922931 0.290840 0.027 Uiso 1 1 calc R U . . .

C13 C 0.63462(14) 0.6258(4) 0.21358(10) 0.0249(4) Uani 1 1 d

H13A H 0.684730 0.660956 0.187525 0.030 Uiso 1 1 calc R U . . .

H13B H 0.624306 0.424569 0.213893 0.030 Uiso 1 1 calc R U . . .

C14 C 0.54103(14) 0.7616(5) 0.17278(10) 0.0267(4) Uani 1 1 d

H14A H 0.489115 0.712676 0.196049 0.032 Uiso 1 1 calc R U . . .

H14B H 0.549277 0.963734 0.175689 0.032 Uiso 1 1 calc R U . . .

C15 C 0.51032(17) 0.6770(6) 0.09442(12) 0.0347(5) Uani 1 1 d

H15A H 0.505000 0.474109 0.091592 0.042 Uiso 1 1 calc R U . . .

H15B H 0.561091 0.733095 0.070757 0.042 Uiso 1 1 calc R U . . .

C16 C 0.41450(17) 0.8021(6) 0.05377(12) 0.0401(6) Uani 1 1 d

H16A H 0.400846 0.749791 0.003052 0.060 Uiso 1 1 calc R U . . .

H16B H 0.418214 1.002872 0.057944 0.060 Uiso 1 1 calc R U . . .
H16C H 0.362736 0.734240 0.074124 0.060 Uiso 1 1 calc R U . . .

loop_

_atom_site_aniso_label
_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12

O1 0.0355(7) 0.0178(6) 0.0135(6) -0.0022(5) 0.0051(5) -0.0074(5)
O2 0.0207(6) 0.0136(5) 0.0114(5) -0.0011(4) 0.0026(4) 0.0003(5)
O3 0.0266(7) 0.0250(7) 0.0245(7) -0.0073(6) 0.0018(5) 0.0057(6)
C1 0.0189(7) 0.0117(7) 0.0119(7) -0.0015(6) 0.0040(6) 0.0005(6)
C2 0.0229(8) 0.0116(7) 0.0131(7) 0.0010(6) 0.0052(6) 0.0010(6)
C3 0.0234(8) 0.0125(7) 0.0129(7) 0.0013(6) 0.0061(6) 0.0006(6)
C4 0.0321(9) 0.0167(8) 0.0147(7) 0.0005(6) 0.0092(7) -0.0002(7)
C5 0.0312(10) 0.0222(9) 0.0197(8) 0.0020(7) 0.0128(7) -0.0009(8)
C6 0.0254(9) 0.0188(8) 0.0249(9) 0.0008(7) 0.0109(7) -0.0031(7)
C7 0.0224(8) 0.0155(8) 0.0185(8) -0.0008(6) 0.0068(6) -0.0008(6)
C8 0.0192(8) 0.0123(7) 0.0143(7) 0.0012(6) 0.0055(6) 0.0015(6)
C9 0.0198(8) 0.0106(7) 0.0106(7) 0.0002(5) 0.0032(6) -0.0002(6)
C10 0.0199(8) 0.0148(7) 0.0171(7) 0.0005(6) 0.0039(6) -0.0001(6)
C11 0.0239(8) 0.0194(8) 0.0173(8) -0.0041(6) 0.0015(6) 0.0005(7)
C12 0.0226(8) 0.0228(9) 0.0199(8) -0.0016(7) 0.0014(7) 0.0016(7)
C13 0.0259(9) 0.0265(10) 0.0192(8) -0.0031(7) 0.0000(7) 0.0020(8)
C14 0.0248(9) 0.0310(10) 0.0208(8) -0.0009(8) -0.0002(7) 0.0021(8)
C15 0.0329(11) 0.0451(13) 0.0218(9) -0.0030(9) -0.0008(8) 0.0048(10)
C16 0.0348(12) 0.0546(15) 0.0247(10) 0.0000(10) -0.0037(9) 0.0035(11)

_geom_special_details

;

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.

;

loop_

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_geom_bond_atom_site_label_2

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 O2 C10 1.357(2) . ?
 O2 C9 1.4261(19) . ?
 O3 C10 1.199(2) . ?
 C1 C1 1.342(3) 3_776 ?
 C1 C2 1.534(2) . ?
 C1 C9 1.553(2) . ?
 C2 C3 1.470(2) . ?
 C3 C8 1.385(2) . ?
 C3 C4 1.394(2) . ?
 C4 C5 1.389(3) . ?
 C4 H4 0.9500 . ?
 C5 C6 1.401(3) . ?
 C5 H5 0.9500 . ?
 C6 C7 1.391(2) . ?
 C6 H6 0.9500 . ?
 C7 C8 1.388(2) . ?
 C7 H7 0.9500 . ?
 C8 C9 1.515(2) . ?
 C9 C9 1.604(3) 3_766 ?
 C10 C11 1.504(2) . ?
 C11 C12 1.521(3) . ?
 C11 H11A 0.9900 . ?
 C11 H11B 0.9900 . ?
 C12 C13 1.521(3) . ?
 C12 H12A 0.9900 . ?
 C12 H12B 0.9900 . ?
 C13 C14 1.520(3) . ?
 C13 H13A 0.9900 . ?
 C13 H13B 0.9900 . ?
 C14 C15 1.518(3) . ?
 C14 H14A 0.9900 . ?
 C14 H14B 0.9900 . ?
 C15 C16 1.522(3) . ?
 C15 H15A 0.9900 . ?
 C15 H15B 0.9900 . ?
 C16 H16A 0.9800 . ?
 C16 H16B 0.9800 . ?
 C16 H16C 0.9800 . ?

loop_

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 C1 C1 C2 126.09(19) 3_776 . ?
 C1 C1 C9 130.20(19) 3_776 . ?
 C2 C1 C9 103.70(13) . . ?
 O1 C2 C3 125.84(16) . . ?
 O1 C2 C1 127.96(16) . . ?
 C3 C2 C1 106.18(14) . . ?
 C8 C3 C4 121.79(17) . . ?
 C8 C3 C2 109.84(15) . . ?
 C4 C3 C2 128.36(16) . . ?
 C5 C4 C3 117.42(17) . . ?
 C5 C4 H4 121.3 . . ?
 C3 C4 H4 121.3 . . ?
 C4 C5 C6 120.55(17) . . ?
 C4 C5 H5 119.7 . . ?
 C6 C5 H5 119.7 . . ?
 C7 C6 C5 121.64(17) . . ?
 C7 C6 H6 119.2 . . ?
 C5 C6 H6 119.2 . . ?
 C8 C7 C6 117.36(16) . . ?
 C8 C7 H7 121.3 . . ?
 C6 C7 H7 121.3 . . ?
 C3 C8 C7 121.08(16) . . ?
 C3 C8 C9 110.77(15) . . ?
 C7 C8 C9 128.13(15) . . ?
 O2 C9 C8 111.76(14) . . ?
 O2 C9 C1 121.35(13) . . ?
 C8 C9 C1 102.17(13) . . ?
 O2 C9 C9 101.40(15) . 3_766 ?
 C8 C9 C9 110.76(16) . 3_766 ?
 C1 C9 C9 109.45(16) . 3_766 ?
 O3 C10 O2 124.16(16) . . ?
 O3 C10 C11 127.15(17) . . ?
 O2 C10 C11 108.68(14) . . ?
 C10 C11 C12 114.17(15) . . ?
 C10 C11 H11A 108.7 . . ?
 C12 C11 H11A 108.7 . . ?

C10 C11 H11B 108.7 . . ?
 C12 C11 H11B 108.7 . . ?
 H11A C11 H11B 107.6 . . ?
 C13 C12 C11 111.52(16) . . ?
 C13 C12 H12A 109.3 . . ?
 C11 C12 H12A 109.3 . . ?
 C13 C12 H12B 109.3 . . ?
 C11 C12 H12B 109.3 . . ?
 H12A C12 H12B 108.0 . . ?
 C14 C13 C12 114.48(17) . . ?
 C14 C13 H13A 108.6 . . ?
 C12 C13 H13A 108.6 . . ?
 C14 C13 H13B 108.6 . . ?
 C12 C13 H13B 108.6 . . ?
 H13A C13 H13B 107.6 . . ?
 C15 C14 C13 112.93(18) . . ?
 C15 C14 H14A 109.0 . . ?
 C13 C14 H14A 109.0 . . ?
 C15 C14 H14B 109.0 . . ?
 C13 C14 H14B 109.0 . . ?
 H14A C14 H14B 107.8 . . ?
 C14 C15 C16 113.4(2) . . ?
 C14 C15 H15A 108.9 . . ?
 C16 C15 H15A 108.9 . . ?
 C14 C15 H15B 108.9 . . ?
 C16 C15 H15B 108.9 . . ?
 H15A C15 H15B 107.7 . . ?
 C15 C16 H16A 109.5 . . ?
 C15 C16 H16B 109.5 . . ?
 H16A C16 H16B 109.5 . . ?
 C15 C16 H16C 109.5 . . ?
 H16A C16 H16C 109.5 . . ?
 H16B C16 H16C 109.5 . . ?

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 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4

_geom_torsion_publ_flag
 C1 C1 C2 O1 -25.9(3) 3_776 ... ?
 C9 C1 C2 O1 155.31(18) ?
 C1 C1 C2 C3 155.4(2) 3_776 ... ?
 C9 C1 C2 C3 -23.40(17) ?
 O1 C2 C3 C8 -168.02(18) ?
 C1 C2 C3 C8 10.72(19) ?
 O1 C2 C3 C4 13.3(3) ?
 C1 C2 C3 C4 -167.99(18) ?
 C8 C3 C4 C5 -3.1(3) ?
 C2 C3 C4 C5 175.43(18) ?
 C3 C4 C5 C6 -0.6(3) ?
 C4 C5 C6 C7 3.0(3) ?
 C5 C6 C7 C8 -1.7(3) ?
 C4 C3 C8 C7 4.5(3) ?
 C2 C3 C8 C7 -174.27(16) ?
 C4 C3 C8 C9 -174.01(16) ?
 C2 C3 C8 C9 7.2(2) ?
 C6 C7 C8 C3 -2.0(3) ?
 C6 C7 C8 C9 176.25(17) ?
 C10 O2 C9 C8 65.35(19) ?
 C10 O2 C9 C1 -55.3(2) ?
 C10 O2 C9 C9 -176.63(15) ... 3_766 ?
 C3 C8 C9 O2 -152.75(14) ?
 C7 C8 C9 O2 28.8(2) ?
 C3 C8 C9 C1 -21.51(18) ?
 C7 C8 C9 C1 160.07(17) ?
 C3 C8 C9 C9 94.98(19) ... 3_766 ?
 C7 C8 C9 C9 -83.4(2) ... 3_766 ?
 C1 C1 C9 O2 -27.2(3) 3_776 ... ?
 C2 C1 C9 O2 151.56(15) ?
 C1 C1 C9 C8 -152.3(2) 3_776 ... ?
 C2 C1 C9 C8 26.42(16) ?
 C1 C1 C9 C9 90.3(3) 3_776 ... 3_766 ?
 C2 C1 C9 C9 -91.01(18) ... 3_766 ?
 C9 O2 C10 O3 -0.1(3) ?
 C9 O2 C10 C11 -179.24(14) ?
 O3 C10 C11 C12 6.0(3) ?
 O2 C10 C11 C12 -174.88(15) ?
 C10 C11 C12 C13 173.26(16) ?
 C11 C12 C13 C14 177.26(17) ?
 C12 C13 C14 C15 175.09(19) ?
 C13 C14 C15 C16 177.5(2) ?

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_refine_diff_density_rms    0.070

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  PBIT_C7_0m.res
  created by SHELXL-2018/3 at 07:54:29 on 20-Aug-2021
REM Old TITL BIT_C_0m in P2(1)/c
REM SHELXT solution in P2(1)/c
REM R1 0.163, Rweak 0.003, Alpha 0.023, Orientation as input
REM Formula found by SHELXT: C16 O3
CELL 0.71073 14.3129 4.8632 19.2705 90.0000 104.8159 90.0000
ZERR 4.000 0.0046 0.0017 0.0068 0.0000 0.0092 0.0000
LATT 1
SYMM -X, 1/2+Y, 1/2-Z
SFAC C H O
UNIT 64 68 12
TEMP -123.150
SIZE 0.55 0.18 0.15

OMIT -3 1 12
OMIT -3 2 12

L.S. 10
BOND $H
ACTA
CONF
LIST 6
FMAP 2
PLAN 20
WGHT 0.065700 1.680100
FVAR 0.64712
O1 3 1.057578 1.048505 0.645817 11.00000 0.03547 0.01785 =
    0.01346 -0.00223 0.00515 -0.00738
O2 3 0.894746 0.540813 0.428942 11.00000 0.02073 0.01364 =
    0.01144 -0.00111 0.00261 0.00030
O3 3 0.780758 0.854932 0.434819 11.00000 0.02656 0.02499 =
    0.02446 -0.00731 0.00180 0.00566
C1 1 0.991838 0.895186 0.520304 11.00000 0.01888 0.01170 =
    0.01185 -0.00153 0.00402 0.00054
C2 1 1.004510 0.901530 0.601802 11.00000 0.02293 0.01163 =
    0.01313 0.00097 0.00515 0.00099

```

C3	1	0.940519	0.686900	0.617426	11.00000	0.02342	0.01253 =
		0.01288	0.00132	0.00614	0.00058		
C4	1	0.913355	0.641642	0.680975	11.00000	0.03212	0.01670 =
		0.01469	0.00047	0.00918	-0.00020		
AFIX	43						
H4	2	0.939869	0.747494	0.722811	11.00000	-1.20000	
AFIX	0						
C5	1	0.846017	0.435771	0.680761	11.00000	0.03123	0.02220 =
		0.01969	0.00205	0.01282	-0.00093		
AFIX	43						
H5	2	0.826246	0.398088	0.723248	11.00000	-1.20000	
AFIX	0						
C6	1	0.807006	0.283209	0.618395	11.00000	0.02539	0.01878 =
		0.02486	0.00076	0.01095	-0.00309		
AFIX	43						
H6	2	0.758770	0.149402	0.618741	11.00000	-1.20000	
AFIX	0						
C7	1	0.837156	0.322818	0.555983	11.00000	0.02239	0.01549 =
		0.01850	-0.00076	0.00682	-0.00083		
AFIX	43						
H7	2	0.811718	0.215523	0.514227	11.00000	-1.20000	
AFIX	0						
C8	1	0.905873	0.525294	0.557138	11.00000	0.01924	0.01234 =
		0.01428	0.00123	0.00548	0.00151		
C9	1	0.954850	0.599359	0.498630	11.00000	0.01978	0.01064 =
		0.01056	0.00017	0.00316	-0.00016		
C10	1	0.810701	0.678579	0.402752	11.00000	0.01985	0.01476 =
		0.01714	0.00054	0.00387	-0.00011		
C11	1	0.763239	0.569414	0.329143	11.00000	0.02387	0.01939 =
		0.01727	-0.00408	0.00153	0.00045		
AFIX	23						
H11A	2	0.746599	0.373643	0.333440	11.00000	-1.20000	
H11B	2	0.810259	0.578039	0.299414	11.00000	-1.20000	
AFIX	0						
C12	1	0.672260	0.723707	0.290666	11.00000	0.02259	0.02277 =
		0.01986	-0.00163	0.00138	0.00158		
AFIX	23						
H12A	2	0.621719	0.695990	0.316751	11.00000	-1.20000	
H12B	2	0.686534	0.922931	0.290840	11.00000	-1.20000	
AFIX	0						
C13	1	0.634615	0.625814	0.213584	11.00000	0.02594	0.02646 =
		0.01920	-0.00310	0.00004	0.00202		
AFIX	23						
H13A	2	0.684730	0.660956	0.187525	11.00000	-1.20000	

H13B 2 0.624306 0.424569 0.213893 11.00000 -1.20000
 AFIX 0
 C14 1 0.541026 0.761592 0.172781 11.00000 0.02482 0.03103 =
 0.02083 -0.00092 -0.00022 0.00211
 AFIX 23
 H14A 2 0.489115 0.712676 0.196049 11.00000 -1.20000
 H14B 2 0.549277 0.963734 0.175689 11.00000 -1.20000
 AFIX 0
 C15 1 0.510317 0.676988 0.094425 11.00000 0.03291 0.04510 =
 0.02177 -0.00301 -0.00079 0.00485
 AFIX 23
 H15A 2 0.505000 0.474109 0.091592 11.00000 -1.20000
 H15B 2 0.561091 0.733095 0.070757 11.00000 -1.20000
 AFIX 0
 C16 1 0.414502 0.802106 0.053768 11.00000 0.03475 0.05456 =
 0.02472 0.00005 -0.00368 0.00345
 AFIX 137
 H16A 2 0.400846 0.749791 0.003052 11.00000 -1.50000
 H16B 2 0.418214 1.002872 0.057944 11.00000 -1.50000
 H16C 2 0.362736 0.734240 0.074124 11.00000 -1.50000
 AFIX 0
 HKLF 4

REM BIT_C_0m_a.res in P2(1)/c

REM wR2 = 0.1693, GooF = S = 1.087, Restrained GooF = 1.087 for all data

REM R1 = 0.0591 for 3000 Fo > 4sig(Fo) and 0.0714 for all 3652 data

REM 173 parameters refined using 0 restraints

END

WGHT 0.0657 1.6805

REM Highest difference peak 0.387, deepest hole -0.305, 1-sigma level 0.070

Q1	1	0.8889	0.3632	0.5612	11.00000	0.05	0.39
Q2	1	0.9719	0.7495	0.5079	11.00000	0.05	0.34
Q3	1	1.0117	0.5646	0.5084	11.00000	0.05	0.31
Q4	1	0.8924	0.4901	0.6822	11.00000	0.05	0.30
Q5	1	1.0513	0.6347	0.7100	11.00000	0.05	0.28
Q6	1	1.0014	0.9129	0.5592	11.00000	0.05	0.28
Q7	1	0.9826	0.7488	0.6194	11.00000	0.05	0.28
Q8	1	0.9649	0.7994	0.6086	11.00000	0.05	0.28
Q9	1	0.7085	0.6008	0.3100	11.00000	0.05	0.27
Q10	1	1.0018	0.7149	0.6267	11.00000	0.05	0.27

Q11	1	0.8534	0.4745	0.5488	11.00000	0.05	0.27
Q12	1	0.8192	0.7532	0.7067	11.00000	0.05	0.27
Q13	1	0.9326	0.6190	0.3694	11.00000	0.05	0.26
Q14	1	0.8504	0.6168	0.6680	11.00000	0.05	0.26
Q15	1	0.9781	1.0716	0.6291	11.00000	0.05	0.25
Q16	1	0.5228	0.7024	0.1328	11.00000	0.05	0.25
Q17	1	1.0546	0.6811	0.6402	11.00000	0.05	0.24
Q18	1	0.8317	0.2853	0.5889	11.00000	0.05	0.24
Q19	1	0.9276	0.5444	0.5293	11.00000	0.05	0.23
Q20	1	0.9305	0.5721	0.5963	11.00000	0.05	0.23

;

_shelx_res_checksum 78449

polyBIT-8 (see CCDC-2088148 for more complete data)

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_shelx_SHELXL_version_number '2018/3'
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_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C17 H19 O3'
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  _atom_type_scatter_dispersion_real
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  _atom_type_scatter_source
  'C' 'C' 0.0033 0.0016
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0106 0.0060
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
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```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
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  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, y+1/2, -z+1/2'
  '-x, -y, -z'
  'x, -y-1/2, z-1/2'
```

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_cell_angle_gamma	90
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_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflms_used	6592
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_exptl_crystal_density_meas	?
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_exptl_crystal_size_mid	0.120
_exptl_crystal_size_min	0.060
_exptl_absorpt_coefficient_mu	0.086
_shelx_estimated_absorpt_T_min	0.945
_shelx_estimated_absorpt_T_max	0.995
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_exptl_absorpt_correction_T_min	0.6514
_exptl_absorpt_correction_T_max	0.7461
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
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_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK α
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_diffn_source_current	'30 mA'
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_diffn_radiation_monochromator	'Triumph curved graphite crystal'

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Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel
array detector (CPAD)
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_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074

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_diffrn_reflns_av_R_equivalents 0.0610
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_diffrn_reflns_limit_h_max      23
_diffrn_reflns_limit_k_min      -6
_diffrn_reflns_limit_k_max      5
_diffrn_reflns_limit_l_min      -27
_diffrn_reflns_limit_l_max      27
_diffrn_reflns_theta_min        2.307
_diffrn_reflns_theta_max        30.568
_diffrn_reflns_theta_full       25.242
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_diffrn_measured_fraction_theta_full 0.989
_diffrn_reflns_Laue_measured_fraction_max 0.947
_diffrn_reflns_Laue_measured_fraction_full 0.989
_diffrn_reflns_point_group_measured_fraction_max 0.947
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_reflns_number_gt               3143
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_reflns_Friedel_fraction_full   .

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```

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.

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_computing_cell_refinement      'SAINT V8.40A (Bruker, 2019)'
_computing_data_reduction      'SAINT V8.40A (Bruker, 2019)'
_computing_structure_solution   'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1146 (Hübschle et al., 2011)
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_computing_molecular_graphics  ?
_computing_publication_material ?
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'w=1/[s^2^(Fo^2)+(0.0510P)^2+0.8366P] where P=(Fo^2+2Fc^2)/3'
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 O2 O 0.09744(6) 0.54117(19) 0.48298(5) 0.01138(19) Uani 1 1 d
 O3 O 0.20245(7) 0.8557(2) 0.54640(6) 0.0207(2) Uani 1 1 d
 C1 C 0.00725(8) 0.8949(3) 0.52450(7) 0.0104(2) Uani 1 1 d
 C2 C -0.00478(9) 0.9028(3) 0.59943(7) 0.0124(2) Uani 1 1 d
 C3 C 0.05428(9) 0.6891(3) 0.64797(7) 0.0125(2) Uani 1 1 d
 C4 C 0.08024(10) 0.6463(3) 0.72545(8) 0.0166(3) Uani 1 1 d
 H4 H 0.056257 0.753629 0.753945 0.020 Uiso 1 1 calc R U . . .
 C5 C 0.14238(10) 0.4413(3) 0.75953(8) 0.0193(3) Uani 1 1 d
 H5 H 0.161065 0.406011 0.812147 0.023 Uiso 1 1 calc R U . . .
 C6 C 0.17762(10) 0.2866(3) 0.71704(8) 0.0183(3) Uani 1 1 d
 H6 H 0.221979 0.153019 0.741845 0.022 Uiso 1 1 calc R U . . .
 C7 C 0.14924(9) 0.3236(3) 0.63906(7) 0.0143(3) Uani 1 1 d
 H7 H 0.172209 0.214211 0.610222 0.017 Uiso 1 1 calc R U . . .
 C8 C 0.08617(9) 0.5263(3) 0.60515(7) 0.0111(2) Uani 1 1 d
 C9 C 0.04152(8) 0.5993(3) 0.52178(7) 0.0099(2) Uani 1 1 d
 C10 C 0.17528(9) 0.6770(3) 0.49964(8) 0.0135(3) Uani 1 1 d
 C11 C 0.22066(9) 0.5603(3) 0.45218(8) 0.0174(3) Uani 1 1 d
 H11A H 0.177828 0.557668 0.398556 0.021 Uiso 1 1 calc R U . . .
 H11B H 0.237911 0.367785 0.467883 0.021 Uiso 1 1 calc R U . . .
 C12 C 0.30285(9) 0.7192(3) 0.45802(8) 0.0180(3) Uani 1 1 d
 H12A H 0.346514 0.719305 0.511372 0.022 Uiso 1 1 calc R U . . .
 H12B H 0.286144 0.912385 0.442760 0.022 Uiso 1 1 calc R U . . .
 C13 C 0.34585(10) 0.5963(3) 0.40825(9) 0.0224(3) Uani 1 1 d
 H13A H 0.360844 0.401706 0.422807 0.027 Uiso 1 1 calc R U . . .
 H13B H 0.302189 0.599492 0.354904 0.027 Uiso 1 1 calc R U . . .
 C14 C 0.42961(10) 0.7459(3) 0.41372(9) 0.0222(3) Uani 1 1 d
 H14A H 0.472516 0.747897 0.467325 0.027 Uiso 1 1 calc R U . . .
 H14B H 0.414224 0.938997 0.397592 0.027 Uiso 1 1 calc R U . . .
 C15 C 0.47431(10) 0.6177(3) 0.36579(9) 0.0241(3) Uani 1 1 d
 H15A H 0.488323 0.423124 0.380940 0.029 Uiso 1 1 calc R U . . .
 H15B H 0.432079 0.620484 0.311979 0.029 Uiso 1 1 calc R U . . .
 C16 C 0.55934(11) 0.7642(4) 0.37332(10) 0.0268(4) Uani 1 1 d
 H16A H 0.601000 0.765079 0.427311 0.032 Uiso 1 1 calc R U . . .
 H16B H 0.545052 0.957679 0.357100 0.032 Uiso 1 1 calc R U . . .
 C17 C 0.60529(13) 0.6332(5) 0.32690(12) 0.0388(5) Uani 1 1 d
 H17A H 0.563580 0.622970 0.273679 0.058 Uiso 1 1 calc R U . . .

H17B H 0.625064 0.447533 0.345969 0.058 Uiso 1 1 calc R U . . .
H17C H 0.656798 0.744625 0.331176 0.058 Uiso 1 1 calc R U . . .

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O2 0.0149(4) 0.0086(4) 0.0146(4) -0.0013(3) 0.0100(4) 0.0001(3)
O3 0.0213(5) 0.0181(6) 0.0266(6) -0.0100(4) 0.0136(4) -0.0049(4)
C1 0.0142(6) 0.0072(6) 0.0119(5) -0.0011(4) 0.0075(5) -0.0003(4)
C2 0.0202(6) 0.0065(6) 0.0136(6) -0.0007(4) 0.0101(5) -0.0014(5)
C3 0.0180(6) 0.0092(6) 0.0125(6) 0.0002(4) 0.0083(5) -0.0007(5)
C4 0.0250(7) 0.0133(7) 0.0140(6) -0.0001(5) 0.0106(5) 0.0004(5)
C5 0.0277(7) 0.0177(7) 0.0124(6) 0.0034(5) 0.0079(6) 0.0022(6)
C6 0.0213(7) 0.0148(7) 0.0170(6) 0.0036(5) 0.0057(5) 0.0033(5)
C7 0.0174(6) 0.0100(6) 0.0161(6) -0.0003(5) 0.0072(5) 0.0008(5)
C8 0.0156(6) 0.0076(6) 0.0111(5) 0.0003(4) 0.0063(5) -0.0009(4)
C9 0.0151(6) 0.0073(6) 0.0114(5) 0.0003(4) 0.0094(5) 0.0009(4)
C10 0.0151(6) 0.0102(6) 0.0173(6) -0.0002(5) 0.0089(5) 0.0001(5)
C11 0.0191(7) 0.0143(7) 0.0239(7) -0.0049(5) 0.0138(6) -0.0004(5)
C12 0.0185(6) 0.0163(7) 0.0241(7) -0.0041(5) 0.0138(6) -0.0021(5)
C13 0.0220(7) 0.0212(8) 0.0318(8) -0.0088(6) 0.0188(6) -0.0046(6)
C14 0.0223(7) 0.0198(8) 0.0315(8) -0.0055(6) 0.0180(6) -0.0044(6)
C15 0.0242(7) 0.0230(8) 0.0329(8) -0.0054(6) 0.0196(7) -0.0036(6)
C16 0.0226(8) 0.0286(9) 0.0352(9) -0.0003(7) 0.0180(7) -0.0031(6)
C17 0.0309(9) 0.0510(12) 0.0475(11) 0.0019(9) 0.0293(9) 0.0017(8)

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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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 O2 C10 1.3580(16) . ?
 O2 C9 1.4258(14) . ?
 O3 C10 1.2020(17) . ?
 C1 C1 1.347(2) 3_576 ?
 C1 C2 1.5346(17) . ?
 C1 C9 1.5528(17) . ?
 C2 C3 1.4727(19) . ?
 C3 C8 1.3890(17) . ?
 C3 C4 1.3946(18) . ?
 C4 C5 1.389(2) . ?
 C4 H4 0.9500 . ?
 C5 C6 1.397(2) . ?
 C5 H5 0.9500 . ?
 C6 C7 1.3953(19) . ?
 C6 H6 0.9500 . ?
 C7 C8 1.3889(19) . ?
 C7 H7 0.9500 . ?
 C8 C9 1.5169(17) . ?
 C9 C9 1.606(3) 3_566 ?
 C10 C11 1.5036(18) . ?
 C11 C12 1.5199(19) . ?
 C11 H11A 0.9900 . ?
 C11 H11B 0.9900 . ?
 C12 C13 1.5230(19) . ?
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 C12 H12B 0.9900 . ?
 C13 C14 1.522(2) . ?
 C13 H13A 0.9900 . ?
 C13 H13B 0.9900 . ?
 C14 C15 1.525(2) . ?
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 C14 H14B 0.9900 . ?
 C15 C16 1.522(2) . ?
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C17 H17B 0.9800 . ?
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C10 O2 C9 121.49(10) . . ?
C1 C1 C2 125.99(14) 3_576 . ?
C1 C1 C9 130.10(14) 3_576 . ?
C2 C1 C9 103.89(10) . . ?
O1 C2 C3 125.83(12) . . ?
O1 C2 C1 127.91(12) . . ?
C3 C2 C1 106.24(10) . . ?
C8 C3 C4 121.66(12) . . ?
C8 C3 C2 109.53(11) . . ?
C4 C3 C2 128.78(12) . . ?
C5 C4 C3 117.69(12) . . ?
C5 C4 H4 121.2 . . ?
C3 C4 H4 121.2 . . ?
C4 C5 C6 120.52(13) . . ?
C4 C5 H5 119.7 . . ?
C6 C5 H5 119.7 . . ?
C7 C6 C5 121.61(13) . . ?
C7 C6 H6 119.2 . . ?
C5 C6 H6 119.2 . . ?
C8 C7 C6 117.54(12) . . ?
C8 C7 H7 121.2 . . ?
C6 C7 H7 121.2 . . ?
C7 C8 C3 120.86(12) . . ?
C7 C8 C9 128.11(11) . . ?
C3 C8 C9 111.01(11) . . ?
O2 C9 C8 111.70(10) . . ?
O2 C9 C1 121.31(10) . . ?
C8 C9 C1 102.06(10) . . ?
O2 C9 C9 101.34(11) . 3_566 ?
C8 C9 C9 111.18(12) . 3_566 ?
C1 C9 C9 109.37(12) . 3_566 ?
O3 C10 O2 124.09(12) . . ?
O3 C10 C11 127.14(12) . . ?

O2 C10 C11 108.75(11) .. ?
 C10 C11 C12 113.87(12) .. ?
 C10 C11 H11A 108.8 .. ?
 C12 C11 H11A 108.8 .. ?
 C10 C11 H11B 108.8 .. ?
 C12 C11 H11B 108.8 .. ?
 H11A C11 H11B 107.7 .. ?
 C11 C12 C13 112.05(12) .. ?
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 C13 C12 H12A 109.2 .. ?
 C11 C12 H12B 109.2 .. ?
 C13 C12 H12B 109.2 .. ?
 H12A C12 H12B 107.9 .. ?
 C14 C13 C12 113.75(12) .. ?
 C14 C13 H13A 108.8 .. ?
 C12 C13 H13A 108.8 .. ?
 C14 C13 H13B 108.8 .. ?
 C12 C13 H13B 108.8 .. ?
 H13A C13 H13B 107.7 .. ?
 C13 C14 C15 113.80(13) .. ?
 C13 C14 H14A 108.8 .. ?
 C15 C14 H14A 108.8 .. ?
 C13 C14 H14B 108.8 .. ?
 C15 C14 H14B 108.8 .. ?
 H14A C14 H14B 107.7 .. ?
 C16 C15 C14 113.37(13) .. ?
 C16 C15 H15A 108.9 .. ?
 C14 C15 H15A 108.9 .. ?
 C16 C15 H15B 108.9 .. ?
 C14 C15 H15B 108.9 .. ?
 H15A C15 H15B 107.7 .. ?
 C15 C16 C17 113.56(15) .. ?
 C15 C16 H16A 108.9 .. ?
 C17 C16 H16A 108.9 .. ?
 C15 C16 H16B 108.9 .. ?
 C17 C16 H16B 108.9 .. ?
 H16A C16 H16B 107.7 .. ?
 C16 C17 H17A 109.5 .. ?
 C16 C17 H17B 109.5 .. ?
 H17A C17 H17B 109.5 .. ?
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 H17B C17 H17C 109.5 .. ?

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 C9 C1 C2 O1 -155.01(14) ... ?
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 C9 C1 C2 C3 23.50(13) ... ?
 O1 C2 C3 C8 167.58(13) ... ?
 C1 C2 C3 C8 -10.97(14) ... ?
 O1 C2 C3 C4 -14.4(2) ... ?
 C1 C2 C3 C4 167.08(13) ... ?
 C8 C3 C4 C5 2.6(2) ... ?
 C2 C3 C4 C5 -175.20(13) ... ?
 C3 C4 C5 C6 0.5(2) ... ?
 C4 C5 C6 C7 -2.8(2) ... ?
 C5 C6 C7 C8 1.8(2) ... ?
 C6 C7 C8 C3 1.34(19) ... ?
 C6 C7 C8 C9 -177.01(13) ... ?
 C4 C3 C8 C7 -3.6(2) ... ?
 C2 C3 C8 C7 174.57(12) ... ?
 C4 C3 C8 C9 174.97(12) ... ?
 C2 C3 C8 C9 -6.82(15) ... ?
 C10 O2 C9 C8 -64.66(14) ... ?
 C10 O2 C9 C1 55.70(16) ... ?
 C10 O2 C9 C9 176.89(11) ... 3_566 ?
 C7 C8 C9 O2 -29.27(18) ... ?
 C3 C8 C9 O2 152.25(11) ... ?
 C7 C8 C9 C1 -160.34(13) ... ?
 C3 C8 C9 C1 21.18(13) ... ?
 C7 C8 C9 C9 83.14(17) ... 3_566 ?
 C3 C8 C9 C9 -95.34(14) ... 3_566 ?
 C1 C1 C9 O2 27.1(2) 3_576 ... ?
 C2 C1 C9 O2 -151.20(11) ... ?
 C1 C1 C9 C8 152.00(18) 3_576 ... ?
 C2 C1 C9 C8 -26.27(12) ... ?
 C1 C1 C9 C9 -90.2(2) 3_576 ... 3_566 ?

C2 C1 C9 C9 91.56(13) . . . 3_566 ?
 C9 O2 C10 O3 -1.1(2) . . . ?
 C9 O2 C10 C11 177.21(11) . . . ?
 O3 C10 C11 C12 -9.2(2) . . . ?
 O2 C10 C11 C12 172.51(12) . . . ?
 C10 C11 C12 C13 -179.20(13) . . . ?
 C11 C12 C13 C14 -178.76(13) . . . ?
 C12 C13 C14 C15 178.21(14) . . . ?
 C13 C14 C15 C16 -178.45(14) . . . ?
 C14 C15 C16 C17 178.74(15) . . . ?

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TITL PBIT_Oct2_0m.res in P2(1)/c

PBIT_Oct2_0m.res

created by SHELXL-2018/3 at 21:14:44 on 16-Oct-2020

REM Old TITL PBIT_Oct2_Tim_0m in P2(1)/c

REM SHELXT solution in P2(1)/c

REM R1 0.148, Rweak 0.004, Alpha 0.035, Orientation as input

REM Formula found by SHELXT: C17 O3

CELL 0.71073 16.4200 4.8651 19.2734 90.000 113.636 90.000

ZERR 4.000 0.0016 0.0004 0.0018 0.000 0.003 0.000

LATT 1

SYMM -X, 1/2+Y, 1/2-Z

SFAC C H O

UNIT 68 76 12

TEMP -123.150

SIZE 0.66 0.12 0.06

L.S. 10

BOND \$H

ACTA

CONF

LIST 6

FMAP 2

PLAN 20

WGHT 0.051000 0.836600

FVAR 0.57626

O1 3 -0.054026 1.049346 0.616091 11.00000 0.02941 0.01380 =
 0.01900 0.00245 0.01707 0.00682

O2	3	0.097439	0.541167	0.482979	11.00000	0.01490	0.00859 =
		0.01458	-0.00135	0.01002	0.00012		
O3	3	0.202451	0.855701	0.546399	11.00000	0.02129	0.01806 =
		0.02659	-0.00996	0.01356	-0.00486		
C1	1	0.007250	0.894913	0.524496	11.00000	0.01418	0.00719 =
		0.01188	-0.00108	0.00748	-0.00030		
C2	1	-0.004779	0.902817	0.599430	11.00000	0.02021	0.00648 =
		0.01360	-0.00066	0.01015	-0.00139		
C3	1	0.054284	0.689139	0.647972	11.00000	0.01804	0.00915 =
		0.01252	0.00025	0.00834	-0.00075		
C4	1	0.080238	0.646326	0.725454	11.00000	0.02503	0.01327 =
		0.01400	-0.00011	0.01058	0.00038		
AFIX	43						
H4	2	0.056257	0.753629	0.753945	11.00000	-1.20000	
AFIX	0						
C5	1	0.142383	0.441303	0.759531	11.00000	0.02773	0.01769 =
		0.01237	0.00343	0.00795	0.00223		
AFIX	43						
H5	2	0.161065	0.406011	0.812147	11.00000	-1.20000	
AFIX	0						
C6	1	0.177616	0.286635	0.717041	11.00000	0.02130	0.01481 =
		0.01701	0.00359	0.00572	0.00326		
AFIX	43						
H6	2	0.221979	0.153019	0.741845	11.00000	-1.20000	
AFIX	0						
C7	1	0.149240	0.323612	0.639059	11.00000	0.01739	0.01004 =
		0.01609	-0.00033	0.00720	0.00082		
AFIX	43						
H7	2	0.172209	0.214211	0.610222	11.00000	-1.20000	
AFIX	0						
C8	1	0.086170	0.526290	0.605154	11.00000	0.01556	0.00763 =
		0.01109	0.00034	0.00635	-0.00094		
C9	1	0.041521	0.599310	0.521784	11.00000	0.01510	0.00726 =
		0.01139	0.00027	0.00940	0.00091		
C10	1	0.175283	0.676970	0.499641	11.00000	0.01511	0.01016 =
		0.01734	-0.00021	0.00885	0.00007		
C11	1	0.220663	0.560277	0.452179	11.00000	0.01912	0.01426 =
		0.02386	-0.00489	0.01379	-0.00045		
AFIX	23						
H11A	2	0.177828	0.557668	0.398556	11.00000	-1.20000	
H11B	2	0.237911	0.367785	0.467883	11.00000	-1.20000	
AFIX	0						
C12	1	0.302854	0.719238	0.458023	11.00000	0.01845	0.01628 =
		0.02414	-0.00411	0.01379	-0.00209		

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AFIX 23
H12A 2 0.346514 0.719305 0.511372 11.00000 -1.20000
H12B 2 0.286144 0.912385 0.442760 11.00000 -1.20000
AFIX 0
C13 1 0.345845 0.596282 0.408246 11.00000 0.02196 0.02117 =
      0.03182 -0.00877 0.01880 -0.00460
AFIX 23
H13A 2 0.360844 0.401706 0.422807 11.00000 -1.20000
H13B 2 0.302189 0.599492 0.354904 11.00000 -1.20000
AFIX 0
C14 1 0.429613 0.745895 0.413722 11.00000 0.02230 0.01979 =
      0.03147 -0.00552 0.01798 -0.00435
AFIX 23
H14A 2 0.472516 0.747897 0.467325 11.00000 -1.20000
H14B 2 0.414224 0.938997 0.397592 11.00000 -1.20000
AFIX 0
C15 1 0.474313 0.617667 0.365793 11.00000 0.02417 0.02300 =
      0.03289 -0.00543 0.01963 -0.00358
AFIX 23
H15A 2 0.488323 0.423124 0.380940 11.00000 -1.20000
H15B 2 0.432079 0.620484 0.311979 11.00000 -1.20000
AFIX 0
C16 1 0.559336 0.764185 0.373318 11.00000 0.02263 0.02862 =
      0.03525 -0.00033 0.01799 -0.00314
AFIX 23
H16A 2 0.601000 0.765079 0.427311 11.00000 -1.20000
H16B 2 0.545052 0.957679 0.357100 11.00000 -1.20000
AFIX 0
C17 1 0.605287 0.633160 0.326902 11.00000 0.03092 0.05097 =
      0.04746 0.00192 0.02928 0.00165
AFIX 137
H17A 2 0.563580 0.622970 0.273679 11.00000 -1.50000
H17B 2 0.625064 0.447533 0.345969 11.00000 -1.50000
H17C 2 0.656798 0.744625 0.331176 11.00000 -1.50000
AFIX 0
HKLF 4

```

```

REM PBIT_Oct2_0m.res in P2(1)/c
REM wR2 = 0.1251, GooF = S = 1.035, Restrained GooF = 1.035 for all data
REM R1 = 0.0494 for 3143 Fo > 4sig(Fo) and 0.0713 for all 4085 data
REM 182 parameters refined using 0 restraints

```

```

END

```

WGHT 0.0508 0.8368

REM Highest difference peak 0.410, deepest hole -0.269, 1-sigma level 0.057

Q1	1	0.0733	0.5706	0.5657	11.00000	0.05	0.41
Q2	1	0.0808	0.6828	0.6860	11.00000	0.05	0.37
Q3	1	0.2041	0.6285	0.4757	11.00000	0.05	0.36
Q4	1	0.0018	0.9272	0.5606	11.00000	0.05	0.35
Q5	1	0.1779	0.3168	0.6803	11.00000	0.05	0.34
Q6	1	0.0778	0.6179	0.6291	11.00000	0.05	0.34
Q7	1	0.1664	0.4001	0.7331	11.00000	0.05	0.27
Q8	1	0.0000	1.0000	0.5000	10.50000	0.05	0.26
Q9	1	0.1302	0.4831	0.6222	11.00000	0.05	0.24
Q10	1	0.0993	0.3638	0.6172	11.00000	0.05	0.23
Q11	1	-0.0340	1.0735	0.6711	11.00000	0.05	0.22
Q12	1	0.5712	0.7229	0.3422	11.00000	0.05	0.22
Q13	1	0.3235	0.6673	0.4305	11.00000	0.05	0.22
Q14	1	0.3920	0.6763	0.4154	11.00000	0.05	0.22
Q15	1	0.0329	0.7556	0.5176	11.00000	0.05	0.22
Q16	1	0.0708	0.5848	0.5069	11.00000	0.05	0.21
Q17	1	0.1618	0.8063	0.5689	11.00000	0.05	0.21
Q18	1	0.2591	0.6674	0.4544	11.00000	0.05	0.21
Q19	1	0.4406	0.7055	0.3818	11.00000	0.05	0.20
Q20	1	0.4465	0.4831	0.3288	11.00000	0.05	0.20

;

_shelx_res_checksum 49205

polyBIT-9 (see CCDC-2133258 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum       'C18 H21 O3'
_chemical_formula_weight    285.35
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  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0181 0.0091
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0492 0.0322
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

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_space_group_crystal_system monoclinic
_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
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```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
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  _space_group_symop_operation_xyz
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  '-x, y+1/2, -z+1/2'
  '-x, -y, -z'
  'x, -y-1/2, z-1/2'
```

_cell_length_a 16.3802(12)
 _cell_length_b 4.8627(3)
 _cell_length_c 19.1505(13)
 _cell_angle_alpha 90
 _cell_angle_beta 99.376(3)
 _cell_angle_gamma 90
 _cell_volume 1505.00(18)
 _cell_formula_units_Z 4
 _cell_measurement_temperature 150(2)
 _cell_measurement_reflms_used 4505
 _cell_measurement_theta_min 4.6805
 _cell_measurement_theta_max 78.9837

_exptl_crystal_description needle
 _exptl_crystal_colour colourless
 _exptl_crystal_density_meas ?
 _exptl_crystal_density_method ?
 _exptl_crystal_density_diffn 1.259
 _exptl_crystal_F_000 612
 _exptl_transmission_factor_min ?
 _exptl_transmission_factor_max ?
 _exptl_crystal_size_max 0.55
 _exptl_crystal_size_mid 0.13
 _exptl_crystal_size_min 0.11
 _exptl_absorpt_coefficient_mu 0.676
 _shelx_estimated_absorpt_T_min ?
 _shelx_estimated_absorpt_T_max ?
 _exptl_absorpt_correction_type multi-scan
 _exptl_absorpt_correction_T_min 0.6133
 _exptl_absorpt_correction_T_max 0.7543
 _exptl_absorpt_process_details

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
 J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details ?
 _diffn_ambient_temperature 150(2)
 _diffn_radiation_wavelength 1.54178
 _diffn_radiation_type CuK\alpha
 _diffn_source 'I-mu-S microsource X-ray tube'
 _diffn_source_current '1.0 mA'
 _diffn_source_voltage '50 kV'
 _diffn_radiation_monochromator 'laterally graded multilayer (Goebel) mirror'

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_diffrn_measurement_device_type
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Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and
photon counting pixel array detector
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_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number           10887
_diffrn_reflns_av_unetI/netI    0.0440
_diffrn_reflns_av_R_equivalents 0.0461
_diffrn_reflns_limit_h_min      -20
_diffrn_reflns_limit_h_max      18
_diffrn_reflns_limit_k_min      -4
_diffrn_reflns_limit_k_max      6
_diffrn_reflns_limit_l_min      -23
_diffrn_reflns_limit_l_max      24
_diffrn_reflns_theta_min        4.680
_diffrn_reflns_theta_max        79.543
_diffrn_reflns_theta_full       67.679
_diffrn_measured_fraction_theta_max 0.947
_diffrn_measured_fraction_theta_full 0.986
_diffrn_reflns_Laue_measured_fraction_max 0.947
_diffrn_reflns_Laue_measured_fraction_full 0.986
_diffrn_reflns_point_group_measured_fraction_max 0.947
_diffrn_reflns_point_group_measured_fraction_full 0.986
_reflns_number_total            3106
_reflns_number_gt               2588
_reflns_threshold_expression     'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

_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.
;

_computing_data_collection      'Apex4 v2021.4-0 (Bruker, 2021)'

```

```

_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction      'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution  'isomorphous replacement'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
;
_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details
;
Solved from another crystal of the same compound, which had been solved
by dual methods (SHELXT (Sheldrick, 2015)).

```

The structure exhibits whole molecule disorder with two alternative sets of atom positions related by a pseudo-mirror operation perpendicular to b. The two disordered moieties were restrained to have similar geometries. Uij components of ADPs for disordered atoms closer to each other than 2.0 Angstrom were restrained to be similar. For the minor moiety, a rigid bond restraint (RIGU) was also applied. Subject to these conditions the occupancy ratio refined to 0.9374(15) to 0.0625(15).

```

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_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type          full
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
'w=1/[\s^2*(Fo^2)+(0.0629P)^2+1.0943P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary    isomor
_atom_sites_solution_secondary  .
_atom_sites_solution_hydrogens  geom
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_refine_ls_extinction_method    none
_refine_ls_extinction_coef      .
_refine_ls_number_reflns        3106
_refine_ls_number_parameters     382
_refine_ls_number_restraints     796
_refine_ls_R_factor_all         0.0599
_refine_ls_R_factor_gt          0.0501
_refine_ls_wR_factor_ref        0.1342
_refine_ls_wR_factor_gt         0.1262
_refine_ls_goodness_of_fit_ref  1.024
_refine_ls_restrained_S_all     0.918
_refine_ls_shift/su_max         0.001

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_refine_ls_shift/su_mean 0.000

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

O1 O 1.04986(8) -0.0503(3) 0.64187(6) 0.0205(3) Uani 0.9374(15) 1 d D U P A 1
O2 O 0.90893(7) 0.4590(2) 0.43675(5) 0.0134(3) Uani 0.9374(15) 1 d D U P A 1
O3 O 0.81328(8) 0.1407(3) 0.45229(7) 0.0232(3) Uani 0.9374(15) 1 d D U P A 1
C1 C 0.99317(10) 0.1042(3) 0.52110(8) 0.0124(3) Uani 0.9374(15) 1 d D U P A 1
C2 C 1.00432(10) 0.0971(3) 0.60216(8) 0.0134(3) Uani 0.9374(15) 1 d D U P A 1
C3 C 0.95004(11) 0.3106(4) 0.62306(8) 0.0144(3) Uani 0.9374(15) 1 d D U P A 1
C4 C 0.92639(11) 0.3549(4) 0.68897(9) 0.0181(4) Uani 0.9374(15) 1 d D U P A 1
H4 H 0.948853 0.247614 0.728974 0.022 Uiso 0.9374(15) 1 calc R U P A 1
C5 C 0.86918(11) 0.5600(4) 0.69410(9) 0.0199(4) Uani 0.9374(15) 1 d D U P A 1
H5 H 0.852013 0.594693 0.738327 0.024 Uiso 0.9374(15) 1 calc R U P A 1
C6 C 0.83636(18) 0.7166(5) 0.63508(12) 0.0194(5) Uani 0.9374(15) 1 d D U P A 1
H6 H 0.795799 0.852301 0.639505 0.023 Uiso 0.9374(15) 1 calc R U P A 1
C7 C 0.86183(10) 0.6779(4) 0.56983(9) 0.0162(4) Uani 0.9374(15) 1 d D U P A 1
H7 H 0.840151 0.787288 0.529994 0.019 Uiso 0.9374(15) 1 calc R U P A 1
C8 C 0.91994(9) 0.4743(3) 0.56506(8) 0.0134(3) Uani 0.9374(15) 1 d D U P A 1
C9 C 0.96150(10) 0.4004(3) 0.50220(9) 0.0123(4) Uani 0.9374(15) 1 d D U P A 1
C10 C 0.83740(10) 0.3184(4) 0.41711(9) 0.0152(4) Uani 0.9374(15) 1 d D U P A 1
C11 C 0.79452(10) 0.4223(4) 0.34663(9) 0.0190(4) Uani 0.9374(15) 1 d D U P A 1
H11A H 0.833256 0.410147 0.312100 0.023 Uiso 0.9374(15) 1 calc R U P A 1
H11B H 0.780364 0.618556 0.351394 0.023 Uiso 0.9374(15) 1 calc R U P A 1
C12 C 0.71605(14) 0.2644(5) 0.31782(11) 0.0218(6) Uani 0.9374(15) 1 d D U P A 1
H12A H 0.729658 0.066944 0.314452 0.026 Uiso 0.9374(15) 1 calc R U P A 1
H12B H 0.676267 0.282222 0.351325 0.026 Uiso 0.9374(15) 1 calc R U P A 1
C13 C 0.67553(11) 0.3665(4) 0.24542(10) 0.0267(4) Uani 0.9374(15) 1 d D U P A 1
H13A H 0.666615 0.567326 0.248102 0.032 Uiso 0.9374(15) 1 calc R U P A 1
H13B H 0.713935 0.334777 0.211303 0.032 Uiso 0.9374(15) 1 calc R U P A 1

C14 C 0.59358(17) 0.2298(10) 0.21756(12) 0.0276(8) Uani 0.9374(15) 1 d D U P A 1
 H14A H 0.602205 0.028510 0.216323 0.033 Uiso 0.9374(15) 1 calc R U P A 1
 H14B H 0.554671 0.266417 0.250978 0.033 Uiso 0.9374(15) 1 calc R U P A 1
 C15 C 0.55424(12) 0.3248(5) 0.14452(11) 0.0331(5) Uani 0.9374(15) 1 d D U P A 1
 H15A H 0.592054 0.280284 0.110724 0.040 Uiso 0.9374(15) 1 calc R U P A 1
 H15B H 0.548099 0.527309 0.145220 0.040 Uiso 0.9374(15) 1 calc R U P A 1
 C16 C 0.4700(2) 0.1977(14) 0.1177(2) 0.0320(6) Uani 0.9374(15) 1 d D U P A 1
 H16A H 0.475789 -0.004984 0.118125 0.038 Uiso 0.9374(15) 1 calc R U P A 1
 H16B H 0.431699 0.246369 0.150784 0.038 Uiso 0.9374(15) 1 calc R U P A 1
 C17 C 0.43210(14) 0.2877(6) 0.04416(12) 0.0423(6) Uani 0.9374(15) 1 d D U P A 1
 H17A H 0.470176 0.236995 0.011072 0.051 Uiso 0.9374(15) 1 calc R U P A 1
 H17B H 0.427119 0.490646 0.043662 0.051 Uiso 0.9374(15) 1 calc R U P A 1
 C18 C 0.34765(18) 0.1652(8) 0.01719(19) 0.0477(9) Uani 0.9374(15) 1 d D U P A 1
 H18A H 0.328902 0.225711 -0.031599 0.072 Uiso 0.9374(15) 1 calc R U P A 1
 H18B H 0.308122 0.226772 0.047210 0.072 Uiso 0.9374(15) 1 calc R U P A 1
 H18C H 0.351374 -0.035889 0.018557 0.072 Uiso 0.9374(15) 1 calc R U P A 1
 O1B O 1.0489(11) 0.549(4) 0.6416(11) 0.032(4) Uani 0.0626(15) 1 d D U P A 2
 O2B O 0.9098(9) 0.039(3) 0.4398(8) 0.017(2) Uani 0.0626(15) 1 d D U P A 2
 O3B O 0.8111(10) 0.339(4) 0.4594(10) 0.021(3) Uani 0.0626(15) 1 d D U P A 2
 C1B C 0.9935(18) 0.398(3) 0.5210(8) 0.018(2) Uani 0.0626(15) 1 d D U P A 2
 C2B C 1.0018(13) 0.408(4) 0.6022(9) 0.017(2) Uani 0.0626(15) 1 d D U P A 2
 C3B C 0.9488(17) 0.194(5) 0.6238(9) 0.018(2) Uani 0.0626(15) 1 d D U P A 2
 C4B C 0.9276(14) 0.136(5) 0.6909(10) 0.020(3) Uani 0.0626(15) 1 d D U P A 2
 H4B H 0.955107 0.227150 0.731991 0.024 Uiso 0.0626(15) 1 calc R U P A 2
 C5B C 0.8667(14) -0.055(5) 0.6962(12) 0.022(4) Uani 0.0626(15) 1 d D U P A 2
 H5B H 0.846010 -0.077717 0.739403 0.026 Uiso 0.0626(15) 1 calc R U P A 2
 C6B C 0.836(3) -0.215(9) 0.6372(14) 0.021(4) Uani 0.0626(15) 1 d D U P A 2
 H6B H 0.795282 -0.351678 0.641372 0.025 Uiso 0.0626(15) 1 calc R U P A 2
 C7B C 0.8625(14) -0.179(5) 0.5725(12) 0.020(4) Uani 0.0626(15) 1 d D U P A 2
 H7B H 0.841678 -0.291472 0.532971 0.024 Uiso 0.0626(15) 1 calc R U P A 2
 C8B C 0.9201(13) 0.023(4) 0.5671(9) 0.018(2) Uani 0.0626(15) 1 d D U P A 2
 C9B C 0.9627(8) 0.099(3) 0.5051(9) 0.017(2) Uani 0.0626(15) 1 d D U P A 2
 C10B C 0.8385(13) 0.183(6) 0.4224(13) 0.016(2) Uani 0.0626(15) 1 d D U P A 2
 C11B C 0.7950(13) 0.068(5) 0.3534(11) 0.022(2) Uani 0.0626(15) 1 d D U P A 2
 H11C H 0.784676 -0.130296 0.359223 0.026 Uiso 0.0626(15) 1 calc R U P A 2
 H11D H 0.831471 0.087735 0.317196 0.026 Uiso 0.0626(15) 1 calc R U P A 2
 C12B C 0.713(2) 0.212(10) 0.3274(18) 0.023(3) Uani 0.0626(15) 1 d D U P A 2
 H12C H 0.673739 0.174256 0.360304 0.028 Uiso 0.0626(15) 1 calc R U P A 2
 H12D H 0.721965 0.413347 0.326143 0.028 Uiso 0.0626(15) 1 calc R U P A 2
 C13B C 0.6775(15) 0.110(6) 0.2541(12) 0.026(3) Uani 0.0626(15) 1 d D U P A 2
 H13C H 0.713698 0.171801 0.220452 0.031 Uiso 0.0626(15) 1 calc R U P A 2
 H13D H 0.677642 -0.093337 0.254292 0.031 Uiso 0.0626(15) 1 calc R U P A 2
 C14B C 0.590(3) 0.210(18) 0.228(2) 0.028(3) Uani 0.0626(15) 1 d D U P A 2
 H14C H 0.553473 0.140814 0.260275 0.033 Uiso 0.0626(15) 1 calc R U P A 2

H14D H 0.589558 0.412976 0.229554 0.033 Uiso 0.0626(15) 1 calc R U P A 2
 C15B C 0.5579(15) 0.115(7) 0.1540(13) 0.030(3) Uani 0.0626(15) 1 d D U P A 2
 H15C H 0.556429 -0.088044 0.153162 0.036 Uiso 0.0626(15) 1 calc R U P A 2
 H15D H 0.596564 0.176058 0.122357 0.036 Uiso 0.0626(15) 1 calc R U P A 2
 C16B C 0.472(4) 0.22(2) 0.125(4) 0.034(3) Uani 0.0626(15) 1 d D U P A 2
 H16C H 0.434527 0.183599 0.159656 0.040 Uiso 0.0626(15) 1 calc R U P A 2
 H16D H 0.474618 0.426085 0.119997 0.040 Uiso 0.0626(15) 1 calc R U P A 2
 C17B C 0.4368(17) 0.099(8) 0.0551(14) 0.034(3) Uani 0.0626(15) 1 d D U P A 2
 H17C H 0.421239 -0.093996 0.062585 0.041 Uiso 0.0626(15) 1 calc R U P A 2
 H17D H 0.480146 0.097536 0.024602 0.041 Uiso 0.0626(15) 1 calc R U P A 2
 C18B C 0.361(3) 0.252(11) 0.016(3) 0.039(6) Uani 0.0626(15) 1 d D U P A 2
 H18D H 0.339362 0.155010 -0.027616 0.059 Uiso 0.0626(15) 1 calc R U P A 2
 H18E H 0.376705 0.439399 0.005436 0.059 Uiso 0.0626(15) 1 calc R U P A 2
 H18F H 0.318141 0.258895 0.046671 0.059 Uiso 0.0626(15) 1 calc R U P A 2

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 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
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 O2 0.0141(5) 0.0136(6) 0.0111(5) 0.0011(4) -0.0021(4) 0.0002(4)
 O3 0.0201(6) 0.0230(8) 0.0244(7) 0.0072(6) -0.0028(5) -0.0050(5)
 C1 0.0113(7) 0.0116(7) 0.0135(7) 0.0015(6) 0.0002(6) -0.0008(6)
 C2 0.0165(7) 0.0091(7) 0.0140(7) -0.0003(6) 0.0005(6) -0.0027(6)
 C3 0.0154(7) 0.0127(8) 0.0148(7) -0.0005(6) 0.0013(6) -0.0009(7)
 C4 0.0212(8) 0.0186(9) 0.0138(7) -0.0002(6) 0.0009(6) -0.0002(7)
 C5 0.0228(8) 0.0201(9) 0.0182(8) -0.0026(7) 0.0071(7) 0.0000(7)
 C6 0.0176(9) 0.0186(13) 0.0224(9) -0.0014(8) 0.0041(7) 0.0032(10)
 C7 0.0147(8) 0.0137(9) 0.0195(8) 0.0016(7) 0.0009(6) -0.0003(7)
 C8 0.0133(7) 0.0118(8) 0.0148(7) -0.0013(6) 0.0017(6) -0.0030(6)
 C9 0.0132(8) 0.0111(8) 0.0111(7) 0.0013(6) -0.0021(6) 0.0004(6)
 C10 0.0146(8) 0.0120(9) 0.0181(8) 0.0020(7) -0.0001(6) 0.0007(7)
 C11 0.0192(8) 0.0186(9) 0.0167(8) 0.0025(6) -0.0041(6) 0.0002(7)
 C12 0.0195(9) 0.0226(13) 0.0211(10) 0.0019(8) -0.0033(7) -0.0008(8)
 C13 0.0237(9) 0.0298(10) 0.0231(9) 0.0027(8) -0.0063(7) -0.0012(8)
 C14 0.0234(10) 0.0300(14) 0.0259(12) 0.0017(12) -0.0064(9) -0.0016(9)
 C15 0.0251(10) 0.0425(13) 0.0277(10) 0.0034(9) -0.0082(8) -0.0023(9)
 C16 0.0234(10) 0.0430(18) 0.0262(14) -0.0002(10) -0.0058(9) -0.0013(11)
 C17 0.0284(11) 0.0638(17) 0.0301(11) 0.0054(11) -0.0091(9) -0.0029(11)
 C18 0.0267(14) 0.076(2) 0.0355(13) 0.0048(16) -0.0102(11) -0.0069(14)

O1B 0.024(7) 0.012(7) 0.052(8) -0.012(7) -0.014(7) 0.000(6)
 O2B 0.013(4) 0.010(4) 0.027(4) -0.002(4) -0.001(4) -0.002(4)
 O3B 0.013(6) 0.017(7) 0.034(7) -0.002(6) 0.007(6) 0.000(5)
 C1B 0.014(4) 0.017(4) 0.021(4) 0.000(4) -0.004(4) 0.002(4)
 C2B 0.016(4) 0.012(4) 0.023(4) -0.001(3) -0.002(4) -0.001(4)
 C3B 0.016(4) 0.014(4) 0.023(4) 0.001(3) -0.003(3) -0.002(4)
 C4B 0.018(5) 0.013(5) 0.029(4) 0.002(4) -0.001(4) -0.004(5)
 C5B 0.015(7) 0.013(7) 0.035(7) 0.002(6) -0.002(6) -0.003(6)
 C6B 0.010(8) 0.017(8) 0.034(8) 0.002(6) 0.000(6) -0.005(7)
 C7B 0.005(6) 0.016(7) 0.038(7) 0.002(6) 0.000(6) -0.003(6)
 C8B 0.012(4) 0.014(4) 0.025(4) 0.000(4) -0.003(4) -0.004(4)
 C9B 0.013(4) 0.016(4) 0.021(4) 0.001(4) -0.002(3) -0.001(4)
 C10B 0.012(4) 0.012(4) 0.022(4) 0.000(4) 0.001(4) -0.002(4)
 C11B 0.019(4) 0.020(4) 0.024(4) -0.001(4) -0.002(4) 0.001(4)
 C12B 0.020(4) 0.024(5) 0.021(4) 0.001(5) -0.008(4) 0.000(4)
 C13B 0.023(4) 0.027(5) 0.024(4) 0.001(4) -0.009(4) 0.002(4)
 C14B 0.022(4) 0.033(5) 0.023(5) 0.001(5) -0.009(4) 0.000(5)
 C15B 0.022(4) 0.037(5) 0.027(4) 0.000(4) -0.008(4) 0.000(4)
 C16B 0.024(5) 0.047(5) 0.026(5) 0.004(5) -0.008(4) 0.001(5)
 C17B 0.024(5) 0.049(6) 0.025(5) 0.007(5) -0.007(5) -0.001(5)
 C18B 0.018(9) 0.062(12) 0.033(10) 0.007(11) -0.009(8) -0.005(10)

_geom_special_details

;

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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loop_

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O2 C10 1.356(2) . ?

O2 C9 1.4290(19) . ?

O3 C10 1.201(2) . ?

C1 C1 1.338(3) 3_756 ?

C1 C2 1.534(2) . ?

C1 C9 1.554(2) . ?
C2 C3 1.465(2) . ?
C3 C8 1.390(2) . ?
C3 C4 1.396(2) . ?
C4 C5 1.383(2) . ?
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C5 C6 1.396(3) . ?
C5 H5 0.9500 . ?
C6 C7 1.393(2) . ?
C6 H6 0.9500 . ?
C7 C8 1.387(2) . ?
C7 H7 0.9500 . ?
C8 C9 1.519(2) . ?
C9 C9 1.603(3) 3_766 ?
C10 C11 1.504(2) . ?
C11 C12 1.522(3) . ?
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C11 H11B 0.9900 . ?
C12 C13 1.520(2) . ?
C12 H12A 0.9900 . ?
C12 H12B 0.9900 . ?
C13 C14 1.515(4) . ?
C13 H13A 0.9900 . ?
C13 H13B 0.9900 . ?
C14 C15 1.514(3) . ?
C14 H14A 0.9900 . ?
C14 H14B 0.9900 . ?
C15 C16 1.523(5) . ?
C15 H15A 0.9900 . ?
C15 H15B 0.9900 . ?
C16 C17 1.509(5) . ?
C16 H16A 0.9900 . ?
C16 H16B 0.9900 . ?
C17 C18 1.517(3) . ?
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C17 H17B 0.9900 . ?
C18 H18A 0.9800 . ?
C18 H18B 0.9800 . ?
C18 H18C 0.9800 . ?
O1B C2B 1.203(16) . ?
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O2B C9B 1.431(16) . ?
O3B C10B 1.180(17) . ?
C1B C1B 1.319(19) 3_766 ?

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 C1B C9B 1.551(16) . ?
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 C3B C8B 1.388(17) . ?
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 C5B H5B 0.9500 . ?
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 C6B H6B 0.9500 . ?
 C7B C8B 1.377(16) . ?
 C7B H7B 0.9500 . ?
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 C9B C9B 1.59(2) 3_756 ?
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 C11B C12B 1.523(19) . ?
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 C11B H11D 0.9900 . ?
 C12B C13B 1.514(19) . ?
 C12B H12C 0.9900 . ?
 C12B H12D 0.9900 . ?
 C13B C14B 1.51(2) . ?
 C13B H13C 0.9900 . ?
 C13B H13D 0.9900 . ?
 C14B C15B 1.50(2) . ?
 C14B H14C 0.9900 . ?
 C14B H14D 0.9900 . ?
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 C15B H15D 0.9900 . ?
 C16B C17B 1.50(2) . ?
 C16B H16C 0.9900 . ?
 C16B H16D 0.9900 . ?
 C17B C18B 1.53(2) . ?
 C17B H17C 0.9900 . ?
 C17B H17D 0.9900 . ?
 C18B H18D 0.9800 . ?
 C18B H18E 0.9800 . ?
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loop_

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 C2 C1 C9 103.71(13) . . ?
 O1 C2 C3 126.00(15) . . ?
 O1 C2 C1 127.54(15) . . ?
 C3 C2 C1 106.44(13) . . ?
 C8 C3 C4 121.32(16) . . ?
 C8 C3 C2 109.86(14) . . ?
 C4 C3 C2 128.79(15) . . ?
 C5 C4 C3 117.80(16) . . ?
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 C3 C4 H4 121.1 . . ?
 C4 C5 C6 120.79(16) . . ?
 C4 C5 H5 119.6 . . ?
 C6 C5 H5 119.6 . . ?
 C7 C6 C5 121.4(2) . . ?
 C7 C6 H6 119.3 . . ?
 C5 C6 H6 119.3 . . ?
 C8 C7 C6 117.67(18) . . ?
 C8 C7 H7 121.2 . . ?
 C6 C7 H7 121.2 . . ?
 C7 C8 C3 120.94(15) . . ?
 C7 C8 C9 128.46(15) . . ?
 C3 C8 C9 110.57(14) . . ?
 O2 C9 C8 111.35(13) . . ?
 O2 C9 C1 121.31(14) . . ?
 C8 C9 C1 102.14(12) . . ?
 O2 C9 C9 101.56(15) . 3_766 ?
 C8 C9 C9 110.91(16) . 3_766 ?
 C1 C9 C9 109.67(16) . 3_766 ?
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 O3 C10 C11 126.63(16) . . ?
 O2 C10 C11 109.63(14) . . ?
 C10 C11 C12 113.55(15) . . ?
 C10 C11 H11A 108.9 . . ?
 C12 C11 H11A 108.9 . . ?
 C10 C11 H11B 108.9 . . ?
 C12 C11 H11B 108.9 . . ?

H11A C11 H11B 107.7 . . ?
C13 C12 C11 112.55(17) . . ?
C13 C12 H12A 109.1 . . ?
C11 C12 H12A 109.1 . . ?
C13 C12 H12B 109.1 . . ?
C11 C12 H12B 109.1 . . ?
H12A C12 H12B 107.8 . . ?
C14 C13 C12 114.1(2) . . ?
C14 C13 H13A 108.7 . . ?
C12 C13 H13A 108.7 . . ?
C14 C13 H13B 108.7 . . ?
C12 C13 H13B 108.7 . . ?
H13A C13 H13B 107.6 . . ?
C15 C14 C13 114.3(3) . . ?
C15 C14 H14A 108.7 . . ?
C13 C14 H14A 108.7 . . ?
C15 C14 H14B 108.7 . . ?
C13 C14 H14B 108.7 . . ?
H14A C14 H14B 107.6 . . ?
C14 C15 C16 114.4(3) . . ?
C14 C15 H15A 108.7 . . ?
C16 C15 H15A 108.7 . . ?
C14 C15 H15B 108.7 . . ?
C16 C15 H15B 108.7 . . ?
H15A C15 H15B 107.6 . . ?
C17 C16 C15 114.2(6) . . ?
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C15 C16 H16A 108.7 . . ?
C17 C16 H16B 108.7 . . ?
C15 C16 H16B 108.7 . . ?
H16A C16 H16B 107.6 . . ?
C16 C17 C18 114.7(4) . . ?
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C18 C17 H17A 108.6 . . ?
C16 C17 H17B 108.6 . . ?
C18 C17 H17B 108.6 . . ?
H17A C17 H17B 107.6 . . ?
C17 C18 H18A 109.5 . . ?
C17 C18 H18B 109.5 . . ?
H18A C18 H18B 109.5 . . ?
C17 C18 H18C 109.5 . . ?
H18A C18 H18C 109.5 . . ?
H18B C18 H18C 109.5 . . ?
C10B O2B C9B 119.1(13) . . ?

C1B C1B C2B 126(2) 3_766 . ?
 C1B C1B C9B 132(2) 3_766 . ?
 C2B C1B C9B 101.6(10) . . ?
 O1B C2B C3B 125.4(18) . . ?
 O1B C2B C1B 126.5(18) . . ?
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 C8B C3B C2B 110.4(13) . . ?
 C4B C3B C2B 130.3(15) . . ?
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 C3B C4B H4B 120.3 . . ?
 C4B C5B C6B 119.2(19) . . ?
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 C5B C6B H6B 119.2 . . ?
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 C6B C7B H7B 120.8 . . ?
 C7B C8B C3B 121.3(15) . . ?
 C7B C8B C9B 129.8(15) . . ?
 C3B C8B C9B 108.9(13) . . ?
 O2B C9B C8B 110.1(13) . . ?
 O2B C9B C1B 119.6(15) . . ?
 C8B C9B C1B 104.5(12) . . ?
 O2B C9B C9B 97.9(14) . 3_756 ?
 C8B C9B C9B 114.2(19) . 3_756 ?
 C1B C9B C9B 111.0(19) . 3_756 ?
 O3B C10B O2B 125.6(17) . . ?
 O3B C10B C11B 126.6(17) . . ?
 O2B C10B C11B 107.0(14) . . ?
 C10B C11B C12B 112.6(16) . . ?
 C10B C11B H11C 109.1 . . ?
 C12B C11B H11C 109.1 . . ?
 C10B C11B H11D 109.1 . . ?
 C12B C11B H11D 109.1 . . ?
 H11C C11B H11D 107.8 . . ?
 C13B C12B C11B 110(2) . . ?
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 C11B C12B H12C 109.7 . . ?
 C13B C12B H12D 109.7 . . ?
 C11B C12B H12D 109.7 . . ?

H12C C12B H12D 108.2 .. ?
 C14B C13B C12B 113.7(19) .. ?
 C14B C13B H13C 108.8 .. ?
 C12B C13B H13C 108.8 .. ?
 C14B C13B H13D 108.8 .. ?
 C12B C13B H13D 108.8 .. ?
 H13C C13B H13D 107.7 .. ?
 C15B C14B C13B 113(2) .. ?
 C15B C14B H14C 109.1 .. ?
 C13B C14B H14C 109.1 .. ?
 C15B C14B H14D 109.1 .. ?
 C13B C14B H14D 109.1 .. ?
 H14C C14B H14D 107.8 .. ?
 C14B C15B C16B 114(2) .. ?
 C14B C15B H15C 108.8 .. ?
 C16B C15B H15C 108.8 .. ?
 C14B C15B H15D 108.8 .. ?
 C16B C15B H15D 108.8 .. ?
 H15C C15B H15D 107.7 .. ?
 C17B C16B C15B 112(2) .. ?
 C17B C16B H16C 109.1 .. ?
 C15B C16B H16C 109.1 .. ?
 C17B C16B H16D 109.1 .. ?
 C15B C16B H16D 109.1 .. ?
 H16C C16B H16D 107.8 .. ?
 C16B C17B C18B 114(3) .. ?
 C16B C17B H17C 108.9 .. ?
 C18B C17B H17C 108.9 .. ?
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 H18D C18B H18E 109.5 .. ?
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 C9 C1 C2 O1 -155.22(17) ?
 C1 C1 C2 C3 -155.4(2) 3_756 ... ?
 C9 C1 C2 C3 23.37(17) ?
 O1 C2 C3 C8 167.90(16) ?
 C1 C2 C3 C8 -10.72(19) ?
 O1 C2 C3 C4 -14.0(3) ?
 C1 C2 C3 C4 167.41(18) ?
 C8 C3 C4 C5 2.7(3) ?
 C2 C3 C4 C5 -175.29(17) ?
 C3 C4 C5 C6 0.1(3) ?
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 C4 C3 C8 C7 -3.5(3) ?
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 C4 C3 C8 C9 174.60(16) ?
 C2 C3 C8 C9 -7.1(2) ?
 C10 O2 C9 C8 -65.91(19) ?
 C10 O2 C9 C1 54.2(2) ?
 C10 O2 C9 C9 175.99(15) ... 3_766 ?
 C7 C8 C9 O2 -29.8(2) ?
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 C7 C8 C9 C1 -160.71(17) ?
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 C3 C8 C9 C9 -95.43(19) ... 3_766 ?
 C1 C1 C9 O2 27.9(3) 3_756 ... ?
 C2 C1 C9 O2 -150.80(13) ?
 C1 C1 C9 C8 152.4(2) 3_756 ... ?
 C2 C1 C9 C8 -26.29(15) ?
 C1 C1 C9 C9 -89.9(3) 3_756 .. 3_766 ?
 C2 C1 C9 C9 91.40(17) ... 3_766 ?
 C9 O2 C10 O3 0.4(3) ?
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 O3 C10 C11 C12 -4.4(3) ?
 O2 C10 C11 C12 176.35(16) ?
 C10 C11 C12 C13 -177.97(17) ?

C11 C12 C13 C14 -175.3(2) ?
 C12 C13 C14 C15 -178.3(2) ?
 C13 C14 C15 C16 -177.3(2) ?
 C14 C15 C16 C17 -178.5(3) ?
 C15 C16 C17 C18 -179.3(3) ?
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 O1B C2B C3B C4B 13(5) ?
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 C6B C7B C8B C3B -2(5) ?
 C6B C7B C8B C9B 177(3) ?
 C4B C3B C8B C7B 9(4) ?
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 C9B O2B C10B O3B -8(5) ?
 C9B O2B C10B C11B -178(2) ?
 O3B C10B C11B C12B 10(6) ?
 O2B C10B C11B C12B 180(3) ?
 C10B C11B C12B C13B 174(3) ?

C11B C12B C13B C14B 172(5) ?
 C12B C13B C14B C15B 178(5) ?
 C13B C14B C15B C16B -177(7) ?
 C14B C15B C16B C17B -173(7) ?
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;

TITL PBIT_C9_2_0m_a.res in P2(1)/c

PBIT_C9_2_0m.res

created by SHELXL-2018/3 at 15:26:39 on 08-Oct-2021

REM Old TITL PBIT_C9_2_0m in P2(1)/c

REM SHELXT solution in P2(1)/c

REM R1 0.139, Rweak 0.002, Alpha 0.022, Orientation as input

REM Formula found by SHELXT: C18 O3

CELL 1.54178 16.3802 4.8627 19.1505 90.000 99.376 90.000

ZERR 4.000 0.0012 0.0003 0.0013 0.000 0.003 0.000

LATT 1

SYMM -X, 1/2+Y, 1/2-Z

SFAC C H O

UNIT 72 84 12

TEMP -123.150

L.S. 10

BOND \$H

ACTA

CONF

LIST 6

FMAP 2

PLAN 20

SIMU 0.01

RIGU O1B > C18B

SADI C1 C1_\$1 C1B C1B_\$2

SADI C9 C9_\$2 C9B C9B_\$1

EQIV \$1 2-x, -y, 1-z

EQIV \$2 2-x, 1-y, 1-z

WGHT 0.062900 1.094300

FVAR 1.05196 0.93736

PART 1 21

O1	3	1.049863	-0.050257	0.641870	21.00000	0.02759	0.01807 =
		0.01443	0.00141	-0.00057	0.00735		
O2	3	0.908927	0.459011	0.436753	21.00000	0.01409	0.01359 =
		0.01112	0.00113	-0.00213	0.00016		
O3	3	0.813279	0.140737	0.452288	21.00000	0.02011	0.02300 =
		0.02437	0.00717	-0.00282	-0.00502		
C1	1	0.993167	0.104226	0.521099	21.00000	0.01134	0.01162 =
		0.01350	0.00148	0.00021	-0.00075		
C2	1	1.004316	0.097069	0.602160	21.00000	0.01655	0.00911 =
		0.01397	-0.00032	0.00049	-0.00265		
C3	1	0.950037	0.310638	0.623058	21.00000	0.01545	0.01270 =
		0.01478	-0.00052	0.00132	-0.00092		
C4	1	0.926389	0.354865	0.688968	21.00000	0.02121	0.01858 =
		0.01382	-0.00018	0.00092	-0.00023		
AFIX	43						
H4	2	0.948853	0.247614	0.728974	21.00000	-1.20000	
AFIX	0						
C5	1	0.869179	0.559963	0.694101	21.00000	0.02281	0.02006 =
		0.01824	-0.00264	0.00711	0.00002		
AFIX	43						
H5	2	0.852013	0.594693	0.738327	21.00000	-1.20000	
AFIX	0						
C6	1	0.836355	0.716560	0.635083	21.00000	0.01756	0.01864 =
		0.02242	-0.00145	0.00413	0.00321		
AFIX	43						
H6	2	0.795799	0.852301	0.639505	21.00000	-1.20000	
AFIX	0						
C7	1	0.861828	0.677945	0.569830	21.00000	0.01468	0.01373 =
		0.01954	0.00160	0.00087	-0.00025		
AFIX	43						
H7	2	0.840151	0.787288	0.529994	21.00000	-1.20000	
AFIX	0						
C8	1	0.919939	0.474296	0.565058	21.00000	0.01335	0.01179 =
		0.01480	-0.00131	0.00167	-0.00295		
C9	1	0.961501	0.400444	0.502204	21.00000	0.01317	0.01113 =
		0.01112	0.00125	-0.00213	0.00036		
C10	1	0.837398	0.318448	0.417113	21.00000	0.01460	0.01200 =
		0.01815	0.00205	-0.00007	0.00073		
C11	1	0.794522	0.422261	0.346633	21.00000	0.01922	0.01858 =
		0.01674	0.00247	-0.00411	0.00021		
AFIX	23						
H11A	2	0.833256	0.410147	0.312100	21.00000	-1.20000	
H11B	2	0.780364	0.618556	0.351394	21.00000	-1.20000	
AFIX	0						

C12	1	0.716046	0.264359	0.317818	21.00000	0.01947	0.02263 =
		0.02115	0.00188	-0.00329	-0.00079		
AFIX	23						
H12A	2	0.729658	0.066944	0.314452	21.00000	-1.20000	
H12B	2	0.676267	0.282222	0.351325	21.00000	-1.20000	
AFIX	0						
C13	1	0.675535	0.366501	0.245416	21.00000	0.02371	0.02984 =
		0.02310	0.00271	-0.00631	-0.00124		
AFIX	23						
H13A	2	0.666615	0.567326	0.248102	21.00000	-1.20000	
H13B	2	0.713935	0.334777	0.211303	21.00000	-1.20000	
AFIX	0						
C14	1	0.593579	0.229842	0.217565	21.00000	0.02342	0.03001 =
		0.02593	0.00169	-0.00642	-0.00162		
AFIX	23						
H14A	2	0.602205	0.028510	0.216323	21.00000	-1.20000	
H14B	2	0.554671	0.266417	0.250978	21.00000	-1.20000	
AFIX	0						
C15	1	0.554239	0.324837	0.144519	21.00000	0.02511	0.04246 =
		0.02768	0.00335	-0.00816	-0.00227		
AFIX	23						
H15A	2	0.592054	0.280284	0.110724	21.00000	-1.20000	
H15B	2	0.548099	0.527309	0.145220	21.00000	-1.20000	
AFIX	0						
C16	1	0.469986	0.197685	0.117705	21.00000	0.02337	0.04304 =
		0.02623	-0.00024	-0.00576	-0.00126		
AFIX	23						
H16A	2	0.475789	-0.004984	0.118125	21.00000	-1.20000	
H16B	2	0.431699	0.246369	0.150784	21.00000	-1.20000	
AFIX	0						
C17	1	0.432097	0.287730	0.044156	21.00000	0.02840	0.06378 =
		0.03011	0.00539	-0.00915	-0.00292		
AFIX	23						
H17A	2	0.470176	0.236995	0.011072	21.00000	-1.20000	
H17B	2	0.427119	0.490646	0.043662	21.00000	-1.20000	
AFIX	0						
C18	1	0.347647	0.165237	0.017194	21.00000	0.02673	0.07571 =
		0.03548	0.00483	-0.01025	-0.00692		
AFIX	137						
H18A	2	0.328902	0.225711	-0.031599	21.00000	-1.50000	
H18B	2	0.308122	0.226772	0.047210	21.00000	-1.50000	
H18C	2	0.351374	-0.035889	0.018557	21.00000	-1.50000	
AFIX	0						
PART 2	-21						

SAME O1 > C18

O1B	3	1.048938	0.548971	0.641615	-21.00000	0.02350	0.01185 =
		0.05250	-0.01160	-0.01382	-0.00037		
O2B	3	0.909792	0.038776	0.439832	-21.00000	0.01297	0.00978 =
		0.02684	-0.00154	-0.00101	-0.00193		
O3B	3	0.811054	0.339406	0.459419	-21.00000	0.01330	0.01705 =
		0.03438	-0.00168	0.00673	-0.00032		
C1B	1	0.993457	0.397530	0.520968	-21.00000	0.01432	0.01689 =
		0.02113	-0.00015	-0.00401	0.00235		
C2B	1	1.001816	0.407980	0.602179	-21.00000	0.01601	0.01216 =
		0.02270	-0.00138	-0.00163	-0.00052		
C3B	1	0.948848	0.194244	0.623779	-21.00000	0.01628	0.01350 =
		0.02274	0.00082	-0.00310	-0.00235		
C4B	1	0.927645	0.135810	0.690938	-21.00000	0.01820	0.01300 =
		0.02850	0.00161	-0.00053	-0.00423		
AFIX	43						
H4B	2	0.955107	0.227150	0.731991	-21.00000	-1.20000	
AFIX	0						
C5B	1	0.866672	-0.055200	0.696195	-21.00000	0.01481	0.01342 =
		0.03451	0.00201	-0.00221	-0.00289		
AFIX	43						
H5B	2	0.846010	-0.077717	0.739403	-21.00000	-1.20000	
AFIX	0						
C6B	1	0.835682	-0.215105	0.637211	-21.00000	0.00988	0.01683 =
		0.03406	0.00248	-0.00042	-0.00497		
AFIX	43						
H6B	2	0.795282	-0.351678	0.641372	-21.00000	-1.20000	
AFIX	0						
C7B	1	0.862540	-0.179273	0.572474	-21.00000	0.00459	0.01619 =
		0.03790	0.00164	-0.00029	-0.00344		
AFIX	43						
H7B	2	0.841678	-0.291472	0.532971	-21.00000	-1.20000	
AFIX	0						
C8B	1	0.920098	0.022840	0.567118	-21.00000	0.01225	0.01432 =
		0.02506	-0.00011	-0.00250	-0.00381		
C9B	1	0.962741	0.098820	0.505104	-21.00000	0.01311	0.01597 =
		0.02054	0.00070	-0.00205	-0.00069		
C10B	1	0.838521	0.182524	0.422369	-21.00000	0.01160	0.01246 =
		0.02179	-0.00011	0.00056	-0.00157		
C11B	1	0.795032	0.068302	0.353379	-21.00000	0.01915	0.02002 =
		0.02437	-0.00119	-0.00233	0.00087		
AFIX	23						
H11C	2	0.784676	-0.130296	0.359223	-21.00000	-1.20000	
H11D	2	0.831471	0.087735	0.317196	-21.00000	-1.20000	

AFIX	0						
C12B	1	0.713120	0.212124	0.327380	-21.00000	0.01983	0.02419 =
		0.02143	0.00143	-0.00846	0.00034		
AFIX	23						
H12C	2	0.673739	0.174256	0.360304	-21.00000	-1.20000	
H12D	2	0.721965	0.413347	0.326143	-21.00000	-1.20000	
AFIX	0						
C13B	1	0.677477	0.110251	0.254054	-21.00000	0.02298	0.02668 =
		0.02394	0.00064	-0.00860	0.00160		
AFIX	23						
H13C	2	0.713698	0.171801	0.220452	-21.00000	-1.20000	
H13D	2	0.677642	-0.093337	0.254292	-21.00000	-1.20000	
AFIX	0						
C14B	1	0.590416	0.209505	0.228057	-21.00000	0.02242	0.03306 =
		0.02350	0.00140	-0.00884	0.00043		
AFIX	23						
H14C	2	0.553473	0.140814	0.260275	-21.00000	-1.20000	
H14D	2	0.589558	0.412976	0.229554	-21.00000	-1.20000	
AFIX	0						
C15B	1	0.557871	0.115476	0.153973	-21.00000	0.02230	0.03652 =
		0.02666	0.00007	-0.00774	-0.00010		
AFIX	23						
H15C	2	0.556429	-0.088044	0.153162	-21.00000	-1.20000	
H15D	2	0.596564	0.176058	0.122357	-21.00000	-1.20000	
AFIX	0						
C16B	1	0.471934	0.223892	0.125230	-21.00000	0.02443	0.04682 =
		0.02571	0.00444	-0.00807	0.00059		
AFIX	23						
H16C	2	0.434527	0.183599	0.159656	-21.00000	-1.20000	
H16D	2	0.474618	0.426085	0.119997	-21.00000	-1.20000	
AFIX	0						
C17B	1	0.436766	0.099210	0.055055	-21.00000	0.02415	0.04944 =
		0.02480	0.00669	-0.00652	-0.00111		
AFIX	23						
H17C	2	0.421239	-0.093996	0.062585	-21.00000	-1.20000	
H17D	2	0.480146	0.097536	0.024602	-21.00000	-1.20000	
AFIX	0						
C18B	1	0.360923	0.251854	0.016412	-21.00000	0.01781	0.06246 =
		0.03253	0.00725	-0.00938	-0.00481		
AFIX	137						
H18D	2	0.339362	0.155010	-0.027616	-21.00000	-1.50000	
H18E	2	0.376705	0.439399	0.005436	-21.00000	-1.50000	
H18F	2	0.318141	0.258895	0.046671	-21.00000	-1.50000	
AFIX	0						

HKLF 4

REM PBIT_C9_2_0m_a.res in P2(1)/c

REM wR2 = 0.1342, GooF = S = 1.024, Restrained GooF = 0.918 for all data

REM R1 = 0.0501 for 2588 Fo > 4sig(Fo) and 0.0599 for all 3106 data

REM 382 parameters refined using 796 restraints

END

WGHT 0.0629 1.0949

REM Highest difference peak 0.414, deepest hole -0.230, 1-sigma level 0.051

Q1	1	0.9761	0.2489	0.5126	11.00000	0.05	0.41
Q2	1	1.0083	0.4589	0.4962	11.00000	0.05	0.32
Q3	1	0.9054	0.6232	0.5728	11.00000	0.05	0.24
Q4	1	0.9240	0.1607	0.6577	11.00000	0.05	0.22
Q5	1	0.9887	0.2492	0.6161	11.00000	0.05	0.22
Q6	1	0.4526	0.4451	0.0239	11.00000	0.05	0.22
Q7	1	0.7594	0.3300	0.3313	11.00000	0.05	0.22
Q8	1	0.9150	0.0677	0.4082	11.00000	0.05	0.21
Q9	1	0.5732	0.4575	0.1209	11.00000	0.05	0.21
Q10	1	0.9263	0.1425	0.7229	11.00000	0.05	0.20
Q11	1	0.4512	0.2782	0.0872	11.00000	0.05	0.20
Q12	1	0.9643	-0.0498	0.5002	11.00000	0.05	0.19
Q13	1	1.0523	0.5354	0.6783	11.00000	0.05	0.18
Q14	1	0.8823	0.4186	0.6903	11.00000	0.05	0.18
Q15	1	0.9241	0.5164	0.6955	11.00000	0.05	0.18
Q16	1	1.0430	0.5979	0.6083	11.00000	0.05	0.18
Q17	1	1.0013	0.0923	0.5566	11.00000	0.05	0.17
Q18	1	0.5086	0.2874	0.1324	11.00000	0.05	0.17
Q19	1	0.9477	0.1544	0.6017	11.00000	0.05	0.17
Q20	1	0.9293	-0.1263	0.6866	11.00000	0.05	0.17

;

_shelx_res_checksum 6960

polyBIT-7B (see CCDC-2131645 for more complete data)

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_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C32 H34 O6'
_chemical_formula_weight    514.59
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loop_
  _atom_type_symbol
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  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0181 0.0091
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0492 0.0322
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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_space_group_name_H-M_alt   'P c a 21'
_space_group_name_Hall      'P 2c -2ac'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, -y, z+1/2'
  'x+1/2, -y, z'
  '-x+1/2, y, z+1/2'
```

```

_cell_length_a      20.031(6)
_cell_length_b      4.8823(16)
_cell_length_c      26.217(8)
_cell_angle_alpha    90
_cell_angle_beta     90
_cell_angle_gamma    90
_cell_volume         2564.0(14)
_cell_formula_units_Z 4
_cell_measurement_temperature 150(2)
_cell_measurement_reflns_used 2415
_cell_measurement_theta_min 4.7424
_cell_measurement_theta_max 78.3509

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_exptl_crystal_colour       colourless
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_exptl_crystal_density_method ?
_exptl_crystal_density_diffn 1.333
_exptl_crystal_F_000        1096
_exptl_transmission_factor_min ?
_exptl_transmission_factor_max ?
_exptl_crystal_size_max      0.110
_exptl_crystal_size_mid      0.040
_exptl_crystal_size_min      0.010
_exptl_absorpt_coefficient_mu 0.737
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_shelx_estimated_absorpt_T_max 0.993
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_exptl_absorpt_correction_T_min 0.5132
_exptl_absorpt_correction_T_max 0.7542
_exptl_absorpt_process_details
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```

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

```

;
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_diffn_radiation_wavelength 1.54178
_diffn_radiation_type       CuK\alpha
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_diffn_source_current        '1.0 mA'
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_diffn_radiation_monochromator 'laterally graded multilayer (Goebel) mirror'

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Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and
photon counting pixel array detector
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_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
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_diffrn_reflns_av_R_equivalents 0.0903
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_diffrn_reflns_limit_k_min       -6
_diffrn_reflns_limit_k_max       4
_diffrn_reflns_limit_l_min       -32
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_diffrn_reflns_theta_min         4.414
_diffrn_reflns_theta_max         78.592
_diffrn_reflns_theta_full        67.679
_diffrn_measured_fraction_theta_max 0.935
_diffrn_measured_fraction_theta_full 0.986
_diffrn_reflns_Laue_measured_fraction_max 0.935
_diffrn_reflns_Laue_measured_fraction_full 0.986
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_diffrn_reflns_point_group_measured_fraction_full 0.900
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_reflns_number_gt                3011
_reflns_threshold_expression      'I > 2\sigma(I)'
_reflns_Friedel_coverage         0.751
_reflns_Friedel_fraction_max     0.736
_reflns_Friedel_fraction_full    0.809

_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.
;

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_computing_structure_solution  'SHELXT (Sheldrick, 2015)'
_computing_structure_refinement
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SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
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_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details        ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type         full
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
'w=1/[s^2^(Fo^2^)+(0.0868P)^2^] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary    dual
_atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens  geom
_refine_ls_hydrogen_treatment   constr
_refine_ls_extinction_method    none
_refine_ls_extinction_coef      .
_refine_ls_abs_structure_details
;
Flack x determined using 890 quotients [(I+)-(I-)]/[(I+)+(I-)]
(Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
;
_refine_ls_abs_structure_Flack  0.1(4)
_chemical_absolute_configuration ukn
_refine_ls_number_reflns        4621
_refine_ls_number_parameters     347
_refine_ls_number_restraints     1
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_refine_ls_R_factor_gt           0.0670
_refine_ls_wR_factor_ref         0.1884
_refine_ls_wR_factor_gt         0.1570
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_refine_ls_shift/su_mean         0.000

loop_
  _atom_site_label
  _atom_site_type_symbol

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_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
O1 O 0.2443(2) 0.2983(11) 0.5309(2) 0.0290(11) Uani 1 1 d . . . . .
O2 O 0.42213(18) -0.2065(10) 0.44233(17) 0.0211(9) Uani 1 1 d . . . . .
O3 O 0.3958(3) 0.1083(13) 0.3825(2) 0.0313(12) Uani 1 1 d . . . . .
O4 O 0.5040(2) 0.2041(11) 0.4664(2) 0.0313(11) Uani 1 1 d . . . . .
O5 O 0.32623(19) 0.7093(10) 0.55387(17) 0.0223(9) Uani 1 1 d . . . . .
O6 O 0.3514(3) 0.3973(14) 0.6154(2) 0.0322(12) Uani 1 1 d . . . . .
C1 C 0.3523(4) 0.1468(16) 0.4943(3) 0.0234(16) Uani 1 1 d . . . . .
C2 C 0.2769(3) 0.1524(14) 0.5025(3) 0.0238(14) Uani 1 1 d . . . . .
C3 C 0.2488(4) -0.0648(18) 0.4688(3) 0.0249(16) Uani 1 1 d . . . . .
C4 C 0.1823(3) -0.1096(16) 0.4556(3) 0.0269(14) Uani 1 1 d . . . . .
H4 H 0.147209 -0.004693 0.470256 0.032 Uiso 1 1 calc R U . . .
C5 C 0.1694(3) -0.3146(17) 0.4202(3) 0.0310(16) Uani 1 1 d . . . . .
H5 H 0.124803 -0.354009 0.410352 0.037 Uiso 1 1 calc R U . . .
C6 C 0.2222(6) -0.4622(19) 0.3990(5) 0.029(2) Uani 1 1 d . . . . .
H6 H 0.212436 -0.595355 0.373626 0.035 Uiso 1 1 calc R U . . .
C7 C 0.2879(4) -0.4240(19) 0.4131(3) 0.0282(15) Uani 1 1 d . . . . .
H7 H 0.323022 -0.529630 0.398704 0.034 Uiso 1 1 calc R U . . .
C8 C 0.3000(3) -0.2229(15) 0.4494(2) 0.0226(14) Uani 1 1 d . . . . .
C9 C 0.3662(3) -0.1488(16) 0.4744(3) 0.0223(15) Uani 1 1 d . . . . .
C10 C 0.3949(4) 0.3559(16) 0.5022(3) 0.0219(15) Uani 1 1 d . . . . .
C11 C 0.4719(3) 0.3508(15) 0.4950(3) 0.0231(14) Uani 1 1 d . . . . .
C12 C 0.4994(4) 0.5627(17) 0.5279(4) 0.0219(15) Uani 1 1 d . . . . .
C13 C 0.5651(3) 0.6059(17) 0.5419(3) 0.0287(15) Uani 1 1 d . . . . .
H13 H 0.600255 0.499488 0.527801 0.034 Uiso 1 1 calc R U . . .
C14 C 0.5781(3) 0.8132(17) 0.5779(3) 0.0319(17) Uani 1 1 d . . . . .
H14 H 0.622765 0.849099 0.587987 0.038 Uiso 1 1 calc R U . . .
C15 C 0.5266(6) 0.9655(19) 0.5988(5) 0.030(2) Uani 1 1 d . . . . .
H15 H 0.536427 1.100363 0.623800 0.036 Uiso 1 1 calc R U . . .
C16 C 0.4597(4) 0.924(2) 0.5837(3) 0.0257(14) Uani 1 1 d . . . . .
H16 H 0.424463 1.029809 0.597910 0.031 Uiso 1 1 calc R U . . .
C17 C 0.4471(3) 0.7235(14) 0.5472(2) 0.0218(13) Uani 1 1 d . . . . .

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C18 C 0.3819(3) 0.6501(16) 0.5221(3) 0.0232(16) Uani 1 1 d
 C19 C 0.4322(4) -0.065(2) 0.3981(3) 0.0242(15) Uani 1 1 d
 C20 C 0.4936(3) -0.1795(16) 0.3727(3) 0.0272(15) Uani 1 1 d
 H20A H 0.481809 -0.355085 0.356078 0.033 Uiso 1 1 calc R U . . .
 H20B H 0.527427 -0.218998 0.399189 0.033 Uiso 1 1 calc R U . . .
 C21 C 0.5249(6) 0.0122(15) 0.3323(5) 0.028(3) Uani 1 1 d
 H21A H 0.494429 0.030351 0.302804 0.033 Uiso 1 1 calc R U . . .
 H21B H 0.531891 0.196493 0.347173 0.033 Uiso 1 1 calc R U . . .
 C22 C 0.5914(3) -0.1060(18) 0.3149(3) 0.0339(17) Uani 1 1 d
 H22A H 0.583148 -0.288184 0.299563 0.041 Uiso 1 1 calc R U . . .
 H22B H 0.619960 -0.134031 0.345303 0.041 Uiso 1 1 calc R U . . .
 C23 C 0.6301(5) 0.067(2) 0.2765(4) 0.035(2) Uani 1 1 d
 H23 H 0.630426 0.261015 0.288967 0.042 Uiso 1 1 calc R U . . .
 C24 C 0.7038(7) -0.034(2) 0.2730(6) 0.049(3) Uani 1 1 d
 H24A H 0.704368 -0.228544 0.263781 0.073 Uiso 1 1 calc R U . . .
 H24B H 0.725778 -0.008874 0.306029 0.073 Uiso 1 1 calc R U . . .
 H24C H 0.727534 0.071738 0.246878 0.073 Uiso 1 1 calc R U . . .
 C25 C 0.5990(7) 0.064(3) 0.2236(6) 0.050(3) Uani 1 1 d
 H25A H 0.622941 0.192725 0.201471 0.075 Uiso 1 1 calc R U . . .
 H25B H 0.552027 0.118089 0.225910 0.075 Uiso 1 1 calc R U . . .
 H25C H 0.602169 -0.120986 0.209292 0.075 Uiso 1 1 calc R U . . .
 C26 C 0.3157(4) 0.576(2) 0.5989(3) 0.0250(14) Uani 1 1 d
 C27 C 0.2542(3) 0.6935(16) 0.6233(3) 0.0284(15) Uani 1 1 d
 H27A H 0.219984 0.721416 0.596490 0.034 Uiso 1 1 calc R U . . .
 H27B H 0.265279 0.875420 0.637623 0.034 Uiso 1 1 calc R U . . .
 C28 C 0.2245(6) 0.5196(16) 0.6649(6) 0.031(3) Uani 1 1 d
 H28A H 0.255163 0.515449 0.694513 0.037 Uiso 1 1 calc R U . . .
 H28B H 0.218751 0.329646 0.652471 0.037 Uiso 1 1 calc R U . . .
 C29 C 0.1566(3) 0.6353(16) 0.6815(3) 0.0280(15) Uani 1 1 d
 H29A H 0.163501 0.827098 0.692583 0.034 Uiso 1 1 calc R U . . .
 H29B H 0.127244 0.640320 0.651143 0.034 Uiso 1 1 calc R U . . .
 C30 C 0.1191(6) 0.4852(18) 0.7241(5) 0.041(3) Uani 1 1 d
 H30 H 0.120802 0.283943 0.717176 0.049 Uiso 1 1 calc R U . . .
 C31 C 0.0478(6) 0.574(3) 0.7248(5) 0.047(2) Uani 1 1 d
 H31A H 0.025961 0.516489 0.693104 0.071 Uiso 1 1 calc R U . . .
 H31B H 0.045661 0.774353 0.727651 0.071 Uiso 1 1 calc R U . . .
 H31C H 0.025013 0.490958 0.753955 0.071 Uiso 1 1 calc R U . . .
 C32 C 0.1521(8) 0.541(3) 0.7767(6) 0.055(3) Uani 1 1 d
 H32A H 0.199458 0.492157 0.775180 0.082 Uiso 1 1 calc R U . . .
 H32B H 0.129841 0.431506 0.802917 0.082 Uiso 1 1 calc R U . . .
 H32C H 0.147725 0.736241 0.785061 0.082 Uiso 1 1 calc R U . . .

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O4 0.026(2) 0.032(3) 0.037(3) -0.006(2) 0.0043(18) 0.003(2)
O5 0.0233(19) 0.022(3) 0.022(2) 0.000(2) 0.0004(17) -0.0005(18)
O6 0.033(3) 0.035(3) 0.029(3) 0.006(3) 0.004(2) 0.007(3)
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C2 0.025(3) 0.022(4) 0.025(3) 0.001(3) -0.001(2) 0.002(3)
C3 0.029(4) 0.026(4) 0.020(4) -0.002(4) -0.001(3) 0.000(4)
C4 0.022(3) 0.026(4) 0.033(3) -0.001(3) -0.002(3) -0.003(3)
C5 0.027(3) 0.030(4) 0.036(4) -0.001(3) -0.006(3) -0.007(3)
C6 0.032(5) 0.029(4) 0.025(5) -0.002(4) -0.006(4) -0.003(3)
C7 0.036(4) 0.023(4) 0.025(4) 0.002(4) -0.003(3) 0.000(4)
C8 0.019(3) 0.027(4) 0.022(3) 0.001(3) -0.001(2) 0.001(2)
C9 0.024(3) 0.024(4) 0.019(3) -0.001(3) 0.003(3) -0.001(3)
C10 0.027(3) 0.022(4) 0.017(3) -0.002(3) -0.001(3) 0.001(3)
C11 0.021(3) 0.022(4) 0.026(3) 0.004(3) 0.002(2) 0.001(3)
C12 0.019(3) 0.021(3) 0.026(4) 0.001(4) 0.003(3) 0.000(3)
C13 0.020(3) 0.033(5) 0.033(4) 0.003(4) 0.000(3) 0.004(3)
C14 0.026(3) 0.036(5) 0.033(4) -0.001(3) -0.007(3) 0.000(3)
C15 0.033(5) 0.030(5) 0.028(5) 0.000(4) -0.007(4) -0.004(3)
C16 0.026(3) 0.030(4) 0.021(3) -0.001(4) -0.003(3) 0.000(4)
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C18 0.013(3) 0.026(4) 0.030(4) 0.003(3) 0.000(3) -0.003(3)
C19 0.020(3) 0.032(4) 0.020(4) 0.003(4) 0.004(3) 0.002(4)
C20 0.033(3) 0.021(4) 0.027(3) -0.001(3) 0.009(3) 0.002(3)
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C24 0.036(5) 0.063(7) 0.048(6) 0.007(5) 0.009(4) 0.007(4)
C25 0.046(6) 0.067(7) 0.037(6) 0.012(6) 0.009(5) 0.002(5)
C26 0.033(4) 0.020(4) 0.022(4) 0.002(4) 0.002(3) 0.000(4)
C27 0.033(3) 0.032(4) 0.020(3) 0.003(3) 0.005(3) 0.006(3)
C28 0.029(6) 0.027(5) 0.038(7) 0.004(3) 0.003(5) 0.005(3)
C29 0.027(3) 0.033(4) 0.024(3) 0.004(3) 0.007(2) 0.001(3)
C30 0.049(5) 0.033(5) 0.040(6) 0.005(4) 0.019(4) 0.000(3)
C31 0.032(4) 0.067(6) 0.042(5) 0.008(6) 0.017(4) -0.014(5)
C32 0.047(6) 0.085(9) 0.032(6) 0.019(6) 0.009(4) 0.011(6)

_geom_special_details

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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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O2 C9 1.429(8) . ?

O3 C19 1.188(11) . ?

O4 C11 1.220(8) . ?

O5 C26 1.363(10) . ?

O5 C18 1.422(9) . ?

O6 C26 1.209(11) . ?

C1 C10 1.347(7) . ?

C1 C2 1.525(9) . ?

C1 C9 1.559(12) . ?

C2 C3 1.490(11) . ?

C3 C8 1.382(11) . ?

C3 C4 1.394(11) . ?

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C4 H4 0.9500 . ?

C5 C6 1.395(14) . ?

C5 H5 0.9500 . ?

C6 C7 1.380(14) . ?

C6 H6 0.9500 . ?

C7 C8 1.389(12) . ?

C7 H7 0.9500 . ?

C8 C9 1.522(9) . ?

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C16 H16 0.9500 . ?
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 O1 C2 C3 125.1(6) . . ?
 O1 C2 C1 128.7(6) . . ?
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 C8 C3 C2 109.6(6) . . ?
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 C1 C10 C11 126.7(5) . . ?
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 O4 C11 C10 127.4(6) . . ?
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 C23 C25 H25B 109.5 . . ?
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 C23 C25 H25C 109.5 . . ?
 H25A C25 H25C 109.5 . . ?
 H25B C25 H25C 109.5 . . ?
 O6 C26 O5 124.3(7) . . ?
 O6 C26 C27 127.4(8) . . ?
 O5 C26 C27 108.3(7) . . ?
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 C27 C28 H28B 109.6 . . ?
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 C28 C29 H29B 107.9 . . ?
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 C30 C31 H31B 109.5 . . ?
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 O1 C2 C3 C8 -168.7(7) ?
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 O1 C2 C3 C4 13.3(14) ?
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 C8 C3 C4 C5 -2.7(12) ?
 C2 C3 C4 C5 175.1(8) ?

C3 C4 C5 C6 -0.8(12) ?
 C4 C5 C6 C7 2.9(15) ?
 C5 C6 C7 C8 -1.4(15) ?
 C4 C3 C8 C7 4.2(13) ?
 C2 C3 C8 C7 -173.9(7) ?
 C4 C3 C8 C9 -174.0(7) ?
 C2 C3 C8 C9 7.9(9) ?
 C6 C7 C8 C3 -2.1(12) ?
 C6 C7 C8 C9 175.8(8) ?
 C19 O2 C9 C8 66.6(9) ?
 C19 O2 C9 C1 -54.4(9) ?
 C19 O2 C9 C18 -175.4(6) . . . 1_545 ?
 C3 C8 C9 O2 -153.6(7) ?
 C7 C8 C9 O2 28.3(11) ?
 C3 C8 C9 C1 -22.1(8) ?
 C7 C8 C9 C1 159.9(8) ?
 C3 C8 C9 C18 94.7(7) . . . 1_545 ?
 C7 C8 C9 C18 -83.4(8) . . . 1_545 ?
 C10 C1 C9 O2 -26.3(9) ?
 C2 C1 C9 O2 152.8(6) ?
 C10 C1 C9 C8 -152.2(6) ?
 C2 C1 C9 C8 26.8(7) ?
 C10 C1 C9 C18 90.1(6) . . . 1_545 ?
 C2 C1 C9 C18 -90.9(5) . . . 1_545 ?
 C2 C1 C10 C18 1.4(10) ?
 C9 C1 C10 C18 -179.8(10) ?
 C2 C1 C10 C11 178.8(8) ?
 C9 C1 C10 C11 -2.3(9) ?
 C1 C10 C11 O4 26.6(10) ?
 C18 C10 C11 O4 -155.4(8) ?
 C1 C10 C11 C12 -155.2(6) ?
 C18 C10 C11 C12 22.8(8) ?
 O4 C11 C12 C13 -14.7(14) ?
 C10 C11 C12 C13 167.1(8) ?
 O4 C11 C12 C17 168.3(7) ?
 C10 C11 C12 C17 -9.9(9) ?
 C17 C12 C13 C14 1.9(12) ?
 C11 C12 C13 C14 -174.7(8) ?
 C12 C13 C14 C15 0.8(13) ?
 C13 C14 C15 C16 -2.0(15) ?
 C14 C15 C16 C17 0.4(14) ?
 C15 C16 C17 C12 2.2(12) ?
 C15 C16 C17 C18 -175.8(8) ?
 C13 C12 C17 C16 -3.5(13) ?

C11 C12 C17 C16 173.7(7) ?
 C13 C12 C17 C18 174.8(7) ?
 C11 C12 C17 C18 -7.9(9) ?
 C26 O5 C18 C17 -65.0(9) ?
 C26 O5 C18 C10 56.7(9) ?
 C26 O5 C18 C9 177.0(6) . . . 1_565 ?
 C16 C17 C18 O5 -28.1(11) ?
 C12 C17 C18 O5 153.8(6) ?
 C16 C17 C18 C10 -159.7(8) ?
 C12 C17 C18 C10 22.1(9) ?
 C16 C17 C18 C9 83.1(8) . . . 1_565 ?
 C12 C17 C18 C9 -95.0(7) . . . 1_565 ?
 C1 C10 C18 O5 25.6(10) ?
 C11 C10 C18 O5 -152.3(6) ?
 C1 C10 C18 C17 151.7(6) ?
 C11 C10 C18 C17 -26.2(7) ?
 C1 C10 C18 C9 -90.0(7) . . . 1_565 ?
 C11 C10 C18 C9 92.1(5) . . . 1_565 ?
 C9 O2 C19 O3 -1.5(14) ?
 C9 O2 C19 C20 -178.3(6) ?
 O3 C19 C20 C21 22.1(15) ?
 O2 C19 C20 C21 -161.2(8) ?
 C19 C20 C21 C22 172.1(8) ?
 C20 C21 C22 C23 -177.5(8) ?
 C21 C22 C23 C25 -72.7(12) ?
 C21 C22 C23 C24 165.0(10) ?
 C18 O5 C26 O6 -1.0(13) ?
 C18 O5 C26 C27 178.3(6) ?
 O6 C26 C27 C28 -17.4(14) ?
 O5 C26 C27 C28 163.3(8) ?
 C26 C27 C28 C29 -171.5(8) ?
 C27 C28 C29 C30 -179.5(9) ?
 C28 C29 C30 C31 -163.9(10) ?
 C28 C29 C30 C32 74.3(11) ?

_refine_diff_density_max 0.346
 _refine_diff_density_min -0.287
 _refine_diff_density_rms 0.066

_shelx_res_file

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TITL PBIT_C6_Me_0m.res in Pca2(1)

PBIT_C6_Me_0m.res

created by SHELXL-2018/3 at 10:05:00 on 20-Sep-2021

REM Old TITL PBIT_C6_Me_0m in Pca2(1)
 REM SHELXT solution in Pca2(1)
 REM R1 0.144, Rweak 0.015, Alpha 0.016, Orientation as input
 REM Flack x = -0.338 (1.317) from Parsons' quotients
 REM Formula found by SHELXT: C32 O6
 CELL 1.54178 20.0310 4.8823 26.2174 90.000 90.000 90.000
 ZERR 4.000 0.0060 0.0016 0.0081 0.000 0.000 0.000
 LATT -1
 SYMM -X, -Y, 1/2+Z
 SYMM 1/2+X, -Y, Z
 SYMM 1/2-X, Y, 1/2+Z
 SFAC C H O
 UNIT 128 136 24
 TEMP -123.150
 SIZE 0.11 0.04 0.01

L.S. 10
 BOND \$H
 ACTA
 CONF
 LIST 6
 FMAP 2
 PLAN 20
 WGHT 0.086800
 FVAR 0.56075
 O1 3 0.244305 0.298349 0.530876 11.00000 0.02499 0.02906 =
 0.03308 -0.00787 0.00707 0.00231
 O2 3 0.422126 -0.206455 0.442331 11.00000 0.01693 0.02398 =
 0.02253 0.00223 0.00377 0.00098
 O3 3 0.395815 0.108284 0.382519 11.00000 0.03252 0.03579 =
 0.02556 0.00787 0.00511 0.00883
 O4 3 0.503975 0.204087 0.466366 11.00000 0.02554 0.03172 =
 0.03676 -0.00631 0.00426 0.00299
 O5 3 0.326230 0.709301 0.553869 11.00000 0.02327 0.02182 =
 0.02167 0.00020 0.00040 -0.00050
 O6 3 0.351372 0.397304 0.615417 11.00000 0.03279 0.03459 =
 0.02915 0.00553 0.00370 0.00678
 C1 1 0.352273 0.146796 0.494282 11.00000 0.02577 0.02406 =
 0.02024 0.00150 -0.00167 -0.00096
 C2 1 0.276886 0.152374 0.502452 11.00000 0.02477 0.02171 =
 0.02496 0.00140 -0.00057 0.00166
 C3 1 0.248784 -0.064803 0.468785 11.00000 0.02851 0.02595 =
 0.02029 -0.00151 -0.00140 0.00033
 C4 1 0.182257 -0.109624 0.455623 11.00000 0.02233 0.02571 =

		0.03264	-0.00124	-0.00168	-0.00335		
AFIX	43						
H4	2	0.147209	-0.004693	0.470256	11.00000	-1.20000	
AFIX	0						
C5	1	0.169438	-0.314559	0.420152	11.00000	0.02716	0.03005 =
		0.03569	-0.00050	-0.00589	-0.00694		
AFIX	43						
H5	2	0.124803	-0.354009	0.410352	11.00000	-1.20000	
AFIX	0						
C6	1	0.222197	-0.462175	0.398969	11.00000	0.03248	0.02904 =
		0.02547	-0.00232	-0.00609	-0.00288		
AFIX	43						
H6	2	0.212436	-0.595355	0.373626	11.00000	-1.20000	
AFIX	0						
C7	1	0.287922	-0.423966	0.413077	11.00000	0.03599	0.02329 =
		0.02547	0.00242	-0.00261	-0.00018		
AFIX	43						
H7	2	0.323022	-0.529630	0.398704	11.00000	-1.20000	
AFIX	0						
C8	1	0.300049	-0.222903	0.449395	11.00000	0.01914	0.02676 =
		0.02183	0.00071	-0.00125	0.00070		
C9	1	0.366199	-0.148751	0.474389	11.00000	0.02407	0.02385 =
		0.01892	-0.00063	0.00348	-0.00139		
C10	1	0.394899	0.355868	0.502216	11.00000	0.02664	0.02205 =
		0.01691	-0.00226	-0.00073	0.00068		
C11	1	0.471858	0.350823	0.494956	11.00000	0.02130	0.02197 =
		0.02591	0.00431	0.00193	0.00129		
C12	1	0.499360	0.562716	0.527855	11.00000	0.01899	0.02078 =
		0.02592	0.00142	0.00349	0.00008		
C13	1	0.565117	0.605918	0.541922	11.00000	0.02002	0.03310 =
		0.03293	0.00279	-0.00046	0.00381		
AFIX	43						
H13	2	0.600255	0.499488	0.527801	11.00000	-1.20000	
AFIX	0						
C14	1	0.578063	0.813177	0.577899	11.00000	0.02620	0.03605 =
		0.03336	-0.00103	-0.00669	-0.00023		
AFIX	43						
H14	2	0.622765	0.849099	0.587987	11.00000	-1.20000	
AFIX	0						
C15	1	0.526556	0.965472	0.598797	11.00000	0.03289	0.02970 =
		0.02768	-0.00046	-0.00690	-0.00400		
AFIX	43						
H15	2	0.536427	1.100363	0.623800	11.00000	-1.20000	
AFIX	0						

C16	1	0.459681	0.924231	0.583681	11.00000	0.02648	0.02955 =
		0.02109	-0.00072	-0.00252	0.00033		
AFIX	43						
H16	2	0.424463	1.029809	0.597910	11.00000	-1.20000	
AFIX	0						
C17	1	0.447087	0.723464	0.547193	11.00000	0.02369	0.01760 =
		0.02408	0.00083	0.00049	0.00212		
C18	1	0.381943	0.650145	0.522115	11.00000	0.01338	0.02571 =
		0.03045	0.00260	-0.00031	-0.00261		
C19	1	0.432169	-0.064791	0.398056	11.00000	0.02027	0.03239 =
		0.02004	0.00273	0.00415	0.00168		
C20	1	0.493599	-0.179455	0.372662	11.00000	0.03280	0.02150 =
		0.02737	-0.00054	0.00862	0.00225		
AFIX	23						
H20A	2	0.481809	-0.355085	0.356078	11.00000	-1.20000	
H20B	2	0.527427	-0.218998	0.399189	11.00000	-1.20000	
AFIX	0						
C21	1	0.524910	0.012240	0.332337	11.00000	0.03050	0.03306 =
		0.01928	0.00062	0.00800	-0.00274		
AFIX	23						
H21A	2	0.494429	0.030351	0.302804	11.00000	-1.20000	
H21B	2	0.531891	0.196493	0.347173	11.00000	-1.20000	
AFIX	0						
C22	1	0.591449	-0.106049	0.314902	11.00000	0.03837	0.02531 =
		0.03810	-0.00195	0.00816	0.00523		
AFIX	23						
H22A	2	0.583148	-0.288184	0.299563	11.00000	-1.20000	
H22B	2	0.619960	-0.134031	0.345303	11.00000	-1.20000	
AFIX	0						
C23	1	0.630054	0.067481	0.276483	11.00000	0.03186	0.03470 =
		0.03827	0.00329	0.00852	0.00460		
AFIX	13						
H23	2	0.630426	0.261015	0.288967	11.00000	-1.20000	
AFIX	0						
C24	1	0.703792	-0.033988	0.272966	11.00000	0.03562	0.06311 =
		0.04808	0.00722	0.00935	0.00734		
AFIX	137						
H24A	2	0.704368	-0.228544	0.263781	11.00000	-1.50000	
H24B	2	0.725778	-0.008874	0.306029	11.00000	-1.50000	
H24C	2	0.727534	0.071738	0.246878	11.00000	-1.50000	
AFIX	0						
C25	1	0.599045	0.064020	0.223595	11.00000	0.04564	0.06734 =
		0.03712	0.01199	0.00886	0.00220		
AFIX	137						

H25A	2	0.622941	0.192725	0.201471	11.00000	-1.50000	
H25B	2	0.552027	0.118089	0.225910	11.00000	-1.50000	
H25C	2	0.602169	-0.120986	0.209292	11.00000	-1.50000	
AFIX	0						
C26	1	0.315733	0.576397	0.598894	11.00000	0.03337	0.02020 =
		0.02151	0.00165	0.00209	0.00009		
C27	1	0.254169	0.693510	0.623262	11.00000	0.03324	0.03166 =
		0.02036	0.00326	0.00484	0.00554		
AFIX	23						
H27A	2	0.219984	0.721416	0.596490	11.00000	-1.20000	
H27B	2	0.265279	0.875420	0.637623	11.00000	-1.20000	
AFIX	0						
C28	1	0.224490	0.519640	0.664914	11.00000	0.02865	0.02731 =
		0.03754	0.00440	0.00335	0.00461		
AFIX	23						
H28A	2	0.255163	0.515449	0.694513	11.00000	-1.20000	
H28B	2	0.218751	0.329646	0.652471	11.00000	-1.20000	
AFIX	0						
C29	1	0.156592	0.635319	0.681512	11.00000	0.02722	0.03287 =
		0.02380	0.00426	0.00663	0.00104		
AFIX	23						
H29A	2	0.163501	0.827098	0.692583	11.00000	-1.20000	
H29B	2	0.127244	0.640320	0.651143	11.00000	-1.20000	
AFIX	0						
C30	1	0.119118	0.485231	0.724112	11.00000	0.04857	0.03290 =
		0.04042	0.00550	0.01851	-0.00010		
AFIX	13						
H30	2	0.120802	0.283943	0.717176	11.00000	-1.20000	
AFIX	0						
C31	1	0.047810	0.574423	0.724762	11.00000	0.03235	0.06726 =
		0.04160	0.00842	0.01737	-0.01375		
AFIX	137						
H31A	2	0.025961	0.516489	0.693104	11.00000	-1.50000	
H31B	2	0.045661	0.774353	0.727651	11.00000	-1.50000	
H31C	2	0.025013	0.490958	0.753955	11.00000	-1.50000	
AFIX	0						
C32	1	0.152072	0.541466	0.776660	11.00000	0.04725	0.08450 =
		0.03215	0.01884	0.00866	0.01124		
AFIX	137						
H32A	2	0.199458	0.492157	0.775180	11.00000	-1.50000	
H32B	2	0.129841	0.431506	0.802917	11.00000	-1.50000	
H32C	2	0.147725	0.736241	0.785061	11.00000	-1.50000	
AFIX	0						
HKLF	4						

REM PBIT_C6_Me_0m.res in Pca2(1)
 REM wR2 = 0.1884, GooF = S = 0.999, Restrained GooF = 0.999 for all data
 REM R1 = 0.0670 for 3011 Fo > 4sig(Fo) and 0.1117 for all 4621 data
 REM 347 parameters refined using 1 restraints

END

WGHT 0.0861 0.0000

REM Highest difference peak 0.346, deepest hole -0.287, 1-sigma level 0.066

Q1	1	0.4733	-0.2182	0.4404	11.00000	0.05	0.35
Q2	1	0.4277	0.3674	0.4932	11.00000	0.05	0.30
Q3	1	0.3277	0.1150	0.5088	11.00000	0.05	0.27
Q4	1	0.2369	0.5482	0.5244	11.00000	0.05	0.26
Q5	1	0.5093	-0.0340	0.4699	11.00000	0.05	0.26
Q6	1	0.2704	0.7349	0.5501	11.00000	0.05	0.26
Q7	1	0.4138	0.6795	0.5331	11.00000	0.05	0.26
Q8	1	0.1980	0.5517	0.7822	11.00000	0.05	0.23
Q9	1	0.3401	0.1541	0.6112	11.00000	0.05	0.23
Q10	1	0.2409	0.5953	0.7709	11.00000	0.05	0.23
Q11	1	0.3090	0.0091	0.4637	11.00000	0.05	0.23
Q12	1	0.4899	-0.4426	0.3854	11.00000	0.05	0.23
Q13	1	0.1525	0.1069	0.4590	11.00000	0.05	0.23
Q14	1	0.5599	0.1411	0.4591	11.00000	0.05	0.22
Q15	1	0.3022	0.3218	0.6002	11.00000	0.05	0.22
Q16	1	0.5654	0.2007	0.5360	11.00000	0.05	0.21
Q17	1	0.4543	-0.3092	0.3907	11.00000	0.05	0.21
Q18	1	0.5331	1.2206	0.6000	11.00000	0.05	0.21
Q19	1	0.1787	0.3775	0.5418	11.00000	0.05	0.21
Q20	1	0.6911	-0.3134	0.2721	11.00000	0.05	0.21

;

_shelx_res_checksum 97829

polyBIT-8D (see CCDC-2131647 for more complete data)

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_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum       'C17 H17 O3'
_chemical_formula_weight    269.30
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loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0181 0.0091
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0492 0.0322
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
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_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
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_shelx_space_group_comment
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```
;
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The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, y+1/2, -z+1/2'
  '-x, -y, -z'
  'x, -y-1/2, z-1/2'
```

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_cell_length_b	4.856(2)
_cell_length_c	19.338(8)
_cell_angle_alpha	90
_cell_angle_beta	101.43(3)
_cell_angle_gamma	90
_cell_volume	1385.3(11)
_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflms_used	2318
_cell_measurement_theta_min	2.9946
_cell_measurement_theta_max	65.1356

_exptl_crystal_description	plate
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.291
_exptl_crystal_F_000	572
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_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.210
_exptl_crystal_size_mid	0.110
_exptl_crystal_size_min	0.020
_exptl_absorpt_coefficient_mu	0.708
_shelx_estimated_absorpt_T_min	0.866
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_exptl_absorpt_correction_T_min	0.4002
_exptl_absorpt_correction_T_max	0.7528
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	1.54178
_diffn_radiation_type	CuK\alpha
_diffn_source	'I-mu-S microsource X-ray tube'
_diffn_source_current	'1.0 mA'
_diffn_source_voltage	'50 kV'
_diffn_radiation_monochromator	'laterally graded multilayer (Goebel) mirror'

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_diffrn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and
photon counting pixel array detector
;
_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number           10182
_diffrn_reflns_av_unetI/netI    0.1436
_diffrn_reflns_av_R_equivalents 0.1495
_diffrn_reflns_limit_h_min      -17
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_diffrn_reflns_limit_k_min      -3
_diffrn_reflns_limit_k_max      5
_diffrn_reflns_limit_l_min      -23
_diffrn_reflns_limit_l_max      21
_diffrn_reflns_theta_min        2.995
_diffrn_reflns_theta_max        66.873
_diffrn_reflns_theta_full       66.873
_diffrn_measured_fraction_theta_max 0.961
_diffrn_measured_fraction_theta_full 0.961
_diffrn_reflns_Laue_measured_fraction_max 0.961
_diffrn_reflns_Laue_measured_fraction_full 0.961
_diffrn_reflns_point_group_measured_fraction_max 0.961
_diffrn_reflns_point_group_measured_fraction_full 0.961
_reflns_number_total            2360
_reflns_number_gt               1297
_reflns_threshold_expression     'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

```

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_reflns_special_details
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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.

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_computing_data_collection      'Apex3 v2019.11-0 (Bruker, 2020)'

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_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction      'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution  'SHELXS-97 (Sheldrick, 2008)'
_computing_structure_refinement
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SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1183 (Hübschle et al., 2011)
;
_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details        ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type         full
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
'w=1/[s^2^(Fo^2^)+(0.1246P)^2+10.2902P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary    direct
_atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens  geom
_refine_ls_hydrogen_treatment   constr
_refine_ls_extinction_method     none
_refine_ls_extinction_coef       .
_refine_ls_number_reflns        2360
_refine_ls_number_parameters     181
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.2054
_refine_ls_R_factor_gt          0.1447
_refine_ls_wR_factor_ref         0.4046
_refine_ls_wR_factor_gt         0.3702
_refine_ls_goodness_of_fit_ref   1.145
_refine_ls_restrained_S_all      1.145
_refine_ls_shift/su_max          0.000
_refine_ls_shift/su_mean         0.000

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_atom_site_U_iso_or_equiv
_atom_site_adp_type
_atom_site_occupancy
_atom_site_site_symmetry_order

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_atom_site_refinement_flags_adp
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_atom_site_disorder_assembly
_atom_site_disorder_group
O1 O -0.0544(4) -0.0487(15) 0.3578(3) 0.0441(17) Uani 1 1 d . . . . .
O2 O 0.0991(4) 0.4589(14) 0.5652(3) 0.0378(16) Uani 1 1 d . . . . .
O3 O 0.2049(4) 0.1456(16) 0.5536(3) 0.0492(18) Uani 1 1 d . . . . .
C1 C 0.0082(6) 0.100(2) 0.4791(4) 0.035(2) Uani 1 1 d . . . . .
C2 C -0.0046(6) 0.0953(19) 0.3990(4) 0.034(2) Uani 1 1 d . . . . .
C3 C 0.0546(6) 0.3163(19) 0.3798(4) 0.035(2) Uani 1 1 d . . . . .
C4 C 0.0819(6) 0.354(2) 0.3152(4) 0.040(2) Uani 1 1 d . . . . .
H4 H 0.058867 0.242347 0.275217 0.047 Uiso 1 1 calc R U . . .
C5 C 0.1448(7) 0.563(2) 0.3127(5) 0.045(2) Uani 1 1 d . . . . .
H5 H 0.163338 0.602651 0.269537 0.054 Uiso 1 1 calc R U . . .
C6 C 0.1801(7) 0.712(2) 0.3722(5) 0.045(2) Uani 1 1 d . . . . .
H6 H 0.226110 0.843243 0.369633 0.054 Uiso 1 1 calc R U . . .
C7 C 0.1518(6) 0.679(2) 0.4365(4) 0.039(2) Uani 1 1 d . . . . .
H7 H 0.174403 0.791723 0.476370 0.047 Uiso 1 1 calc R U . . .
C8 C 0.0876(6) 0.4695(18) 0.4382(4) 0.0316(19) Uani 1 1 d . . . . .
C9 C 0.0413(6) 0.4014(19) 0.4989(4) 0.034(2) Uani 1 1 d . . . . .
C10 C 0.1785(6) 0.324(2) 0.5865(4) 0.038(2) Uani 1 1 d . . . . .
C11 C 0.2260(6) 0.437(2) 0.6562(5) 0.047(2) Uani 1 1 d . . . . .
H11A H 0.181588 0.453970 0.687478 0.056 Uiso 1 1 calc R U . . .
H11B H 0.248190 0.624811 0.648716 0.056 Uiso 1 1 calc R U . . .
C12 C 0.3055(6) 0.264(2) 0.6933(4) 0.047(3) Uani 1 1 d . . . . .
H12A H 0.284485 0.074221 0.699391 0.056 Uiso 1 1 calc R U . . .
H12B H 0.352004 0.255436 0.663617 0.056 Uiso 1 1 calc R U . . .
C13 C 0.3476(7) 0.383(3) 0.7648(5) 0.055(3) Uani 1 1 d . . . . .
H13A H 0.371222 0.568421 0.757614 0.067 Uiso 1 1 calc R U . A 1
H13B H 0.299138 0.405110 0.792347 0.067 Uiso 1 1 calc R U . A 1
C14 C 0.4223(7) 0.218(3) 0.8079(5) 0.055(3) Uani 1 1 d . . . B 1
H14A H 0.470973 0.196715 0.780516 0.066 Uiso 1 1 calc R U . B 1
H14B H 0.398813 0.031915 0.814852 0.066 Uiso 1 1 calc R U . B 1
C15 C 0.4635(8) 0.336(3) 0.8791(5) 0.063(3) Uani 1 1 d . . . B 1
H15A H 0.492047 0.514363 0.871970 0.075 Uiso 1 1 calc R U . B 1
H15B H 0.414142 0.372797 0.904888 0.075 Uiso 1 1 calc R U . B 1
C16 C 0.5313(8) 0.161(3) 0.9233(5) 0.064(3) Uani 1 1 d . . . B 1
H16 H 0.511348 -0.017665 0.932993 0.077 Uiso 1 1 calc R U . B 1
C17 C 0.6139(8) 0.218(4) 0.9508(6) 0.085(5) Uani 1 1 d . . . B 1
H17A H 0.638207 0.393440 0.943232 0.102 Uiso 1 1 calc R U . B 1
H17B H 0.650877 0.084368 0.978719 0.102 Uiso 1 1 calc R U . B 1

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  _atom_site_aniso_U_22
  _atom_site_aniso_U_33
  _atom_site_aniso_U_23
  _atom_site_aniso_U_13
  _atom_site_aniso_U_12
O1 0.061(4) 0.040(4) 0.031(3) -0.001(3) 0.008(3) -0.005(3)
O2 0.041(3) 0.044(4) 0.028(3) -0.002(3) 0.005(2) -0.004(3)
O3 0.049(4) 0.053(5) 0.046(4) -0.014(3) 0.010(3) -0.001(3)
C1 0.041(5) 0.034(6) 0.030(4) -0.001(4) 0.008(3) -0.003(4)
C2 0.052(5) 0.024(5) 0.026(4) -0.004(4) 0.006(4) 0.000(4)
C3 0.054(5) 0.023(5) 0.031(4) 0.000(4) 0.013(4) -0.005(4)
C4 0.062(6) 0.026(5) 0.035(4) 0.000(4) 0.020(4) -0.003(4)
C5 0.062(6) 0.043(6) 0.034(5) 0.001(4) 0.020(4) 0.005(5)
C6 0.053(6) 0.043(6) 0.045(5) 0.004(5) 0.022(4) -0.007(5)
C7 0.052(5) 0.040(6) 0.030(4) -0.009(4) 0.017(4) -0.003(4)
C8 0.046(5) 0.022(5) 0.031(4) 0.000(4) 0.015(3) 0.001(4)
C9 0.049(5) 0.028(6) 0.026(4) -0.002(4) 0.009(3) -0.002(4)
C10 0.038(5) 0.044(6) 0.032(4) -0.001(4) 0.009(4) -0.007(4)
C11 0.053(6) 0.047(7) 0.040(5) -0.010(5) 0.007(4) 0.010(5)
C12 0.046(5) 0.060(7) 0.033(5) 0.001(4) 0.006(4) 0.007(5)
C13 0.048(5) 0.066(8) 0.048(6) -0.012(5) -0.003(4) 0.006(5)
C14 0.060(6) 0.064(8) 0.039(5) -0.003(5) 0.006(4) 0.008(6)
C15 0.071(7) 0.070(9) 0.045(6) -0.014(6) 0.006(5) 0.008(6)
C16 0.066(7) 0.083(10) 0.043(5) 0.005(6) 0.007(5) 0.009(7)
C17 0.064(8) 0.121(14) 0.066(8) 0.024(8) 0.003(6) -0.016(8)

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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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  _geom_bond_atom_site_label_2
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  _geom_bond_site_symmetry_2

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_geom_bond_publ_flag

O1 C2 1.203(10) . ?
 O2 C10 1.353(11) . ?
 O2 C9 1.428(10) . ?
 O3 C10 1.190(11) . ?
 C1 C1 1.321(18) 3_556 ?
 C1 C2 1.522(11) . ?
 C1 C9 1.567(13) . ?
 C2 C3 1.489(12) . ?
 C3 C8 1.361(12) . ?
 C3 C4 1.402(11) . ?
 C4 C5 1.396(13) . ?
 C4 H4 0.9500 . ?
 C5 C6 1.374(13) . ?
 C5 H5 0.9500 . ?
 C6 C7 1.401(11) . ?
 C6 H6 0.9500 . ?
 C7 C8 1.410(12) . ?
 C7 H7 0.9500 . ?
 C8 C9 1.515(11) . ?
 C9 C9 1.578(17) 3_566 ?
 C10 C11 1.500(12) . ?
 C11 C12 1.521(13) . ?
 C11 H11A 0.9900 . ?
 C11 H11B 0.9900 . ?
 C12 C13 1.515(13) . ?
 C12 H12A 0.9900 . ?
 C12 H12B 0.9900 . ?
 C13 C14 1.492(14) . ?
 C13 H13A 0.9900 . ?
 C13 H13B 0.9900 . ?
 C14 C15 1.506(13) . ?
 C14 H14A 0.9900 . ?
 C14 H14B 0.9900 . ?
 C15 C16 1.464(16) . ?
 C15 H15A 0.9900 . ?
 C15 H15B 0.9900 . ?
 C16 C17 1.282(16) . ?
 C16 H16 0.9500 . ?
 C17 H17A 0.9500 . ?
 C17 H17B 0.9500 . ?

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 C10 O2 C9 121.5(7) . . ?
 C1 C1 C2 127.6(11) 3_556 . ?
 C1 C1 C9 128.8(10) 3_556 . ?
 C2 C1 C9 103.5(7) . . ?
 O1 C2 C3 125.4(7) . . ?
 O1 C2 C1 128.2(8) . . ?
 C3 C2 C1 106.3(7) . . ?
 C8 C3 C4 123.1(8) . . ?
 C8 C3 C2 109.0(7) . . ?
 C4 C3 C2 127.6(8) . . ?
 C5 C4 C3 116.4(8) . . ?
 C5 C4 H4 121.8 . . ?
 C3 C4 H4 121.8 . . ?
 C6 C5 C4 120.4(8) . . ?
 C6 C5 H5 119.8 . . ?
 C4 C5 H5 119.8 . . ?
 C5 C6 C7 123.4(9) . . ?
 C5 C6 H6 118.3 . . ?
 C7 C6 H6 118.3 . . ?
 C6 C7 C8 115.6(8) . . ?
 C6 C7 H7 122.2 . . ?
 C8 C7 H7 122.2 . . ?
 C3 C8 C7 121.0(7) . . ?
 C3 C8 C9 112.0(7) . . ?
 C7 C8 C9 126.8(7) . . ?
 O2 C9 C8 111.2(7) . . ?
 O2 C9 C1 120.6(7) . . ?
 C8 C9 C1 100.8(6) . . ?
 O2 C9 C9 101.5(8) . 3_566 ?
 C8 C9 C9 112.0(9) . 3_566 ?
 C1 C9 C9 111.0(9) . 3_566 ?
 O3 C10 O2 124.0(8) . . ?
 O3 C10 C11 126.7(9) . . ?
 O2 C10 C11 109.3(8) . . ?
 C10 C11 C12 114.4(8) . . ?
 C10 C11 H11A 108.7 . . ?
 C12 C11 H11A 108.7 . . ?
 C10 C11 H11B 108.7 . . ?

C12 C11 H11B 108.7 . . ?
 H11A C11 H11B 107.6 . . ?
 C13 C12 C11 111.6(9) . . ?
 C13 C12 H12A 109.3 . . ?
 C11 C12 H12A 109.3 . . ?
 C13 C12 H12B 109.3 . . ?
 C11 C12 H12B 109.3 . . ?
 H12A C12 H12B 108.0 . . ?
 C14 C13 C12 116.0(10) . . ?
 C14 C13 H13A 108.3 . . ?
 C12 C13 H13A 108.3 . . ?
 C14 C13 H13B 108.3 . . ?
 C12 C13 H13B 108.3 . . ?
 H13A C13 H13B 107.4 . . ?
 C13 C14 C15 115.8(10) . . ?
 C13 C14 H14A 108.3 . . ?
 C15 C14 H14A 108.3 . . ?
 C13 C14 H14B 108.3 . . ?
 C15 C14 H14B 108.3 . . ?
 H14A C14 H14B 107.4 . . ?
 C16 C15 C14 115.4(11) . . ?
 C16 C15 H15A 108.4 . . ?
 C14 C15 H15A 108.4 . . ?
 C16 C15 H15B 108.4 . . ?
 C14 C15 H15B 108.4 . . ?
 H15A C15 H15B 107.5 . . ?
 C17 C16 C15 128.7(15) . . ?
 C17 C16 H16 115.7 . . ?
 C15 C16 H16 115.7 . . ?
 C16 C17 H17A 120.0 . . ?
 C16 C17 H17B 120.0 . . ?
 H17A C17 H17B 120.0 . . ?

loop_

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 _geom_torsion_site_symmetry_3
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C1 C1 C2 O1 23.9(18) 3_556 ... ?
 C9 C1 C2 O1 -153.8(10) ... ?
 C1 C1 C2 C3 -158.5(12) 3_556 ... ?
 C9 C1 C2 C3 23.8(9) ... ?
 O1 C2 C3 C8 168.4(9) ... ?
 C1 C2 C3 C8 -9.3(10) ... ?
 O1 C2 C3 C4 -17.8(16) ... ?
 C1 C2 C3 C4 164.6(9) ... ?
 C8 C3 C4 C5 -1.1(14) ... ?
 C2 C3 C4 C5 -174.1(9) ... ?
 C3 C4 C5 C6 3.1(14) ... ?
 C4 C5 C6 C7 -4.6(16) ... ?
 C5 C6 C7 C8 3.8(15) ... ?
 C4 C3 C8 C7 0.5(14) ... ?
 C2 C3 C8 C7 174.7(8) ... ?
 C4 C3 C8 C9 175.6(9) ... ?
 C2 C3 C8 C9 -10.3(11) ... ?
 C6 C7 C8 C3 -1.7(13) ... ?
 C6 C7 C8 C9 -176.0(9) ... ?
 C10 O2 C9 C8 -62.9(10) ... ?
 C10 O2 C9 C1 54.7(11) ... ?
 C10 O2 C9 C9 177.8(8) ... 3_566 ?
 C3 C8 C9 O2 153.6(8) ... ?
 C7 C8 C9 O2 -31.7(12) ... ?
 C3 C8 C9 C1 24.5(9) ... ?
 C7 C8 C9 C1 -160.8(8) ... ?
 C3 C8 C9 C9 -93.6(11) ... 3_566 ?
 C7 C8 C9 C9 81.1(12) ... 3_566 ?
 C1 C1 C9 O2 31.5(16) 3_556 ... ?
 C2 C1 C9 O2 -150.8(7) ... ?
 C1 C1 C9 C8 154.3(12) 3_556 ... ?
 C2 C1 C9 C8 -28.1(8) ... ?
 C1 C1 C9 C9 -86.9(14) 3_556 ... 3_566 ?
 C2 C1 C9 C9 90.8(9) ... 3_566 ?
 C9 O2 C10 O3 -2.4(13) ... ?
 C9 O2 C10 C11 177.3(8) ... ?
 O3 C10 C11 C12 -12.9(15) ... ?
 O2 C10 C11 C12 167.4(8) ... ?
 C10 C11 C12 C13 -177.3(9) ... ?
 C11 C12 C13 C14 176.1(10) ... ?
 C12 C13 C14 C15 -179.6(10) ... ?
 C13 C14 C15 C16 174.9(11) ... ?
 C14 C15 C16 C17 122.8(15) ... ?

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_refine_diff_density_rms    0.104

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  PBIT_Oct2_D_2_0m.res
  created by SHELXL-2018/3 at 17:43:01 on 22-Feb-2021
REM Old TITL PBIT_Oct2_D_2_0m in P2(1)/c
REM SHELXT solution in P2(1)/c
REM R1 0.268, Rweak 0.031, Alpha 0.136, Orientation as input
REM Formula found by SHELXT: C16 O4
CELL 1.54178 15.0529 4.8555 19.3379 90.0000 101.4334 90.0000
ZERR 4.000 0.0075 0.0023 0.0082 0.0000 0.0308 0.0000
LATT 1
SYMM -X, 1/2+Y, 1/2-Z
SFAC C H O
UNIT 68 68 12
TEMP -123.150
SIZE 0.21 0.11 0.02
L.S. 10
BOND $H
CONF
ACTA
LIST 6
FMAP 2
PLAN 20
REM DAMP 200
WGHT 0.124600 10.290200
FVAR 0.54044
O1 3 -0.054386 -0.048661 0.357824 11.00000 0.06076 0.04024 =
    0.03108 -0.00096 0.00838 -0.00482
O2 3 0.099078 0.458877 0.565210 11.00000 0.04087 0.04368 =
    0.02828 -0.00181 0.00545 -0.00403
O3 3 0.204909 0.145607 0.553572 11.00000 0.04864 0.05278 =
    0.04629 -0.01439 0.00976 -0.00084
C1 1 0.008229 0.100236 0.479055 11.00000 0.04105 0.03370 =
    0.03029 -0.00066 0.00801 -0.00289
C2 1 -0.004595 0.095282 0.398967 11.00000 0.05157 0.02418 =
    0.02619 -0.00421 0.00586 -0.00038
C3 1 0.054647 0.316350 0.379784 11.00000 0.05366 0.02348 =
    0.03086 0.00022 0.01311 -0.00517
C4 1 0.081937 0.353574 0.315159 11.00000 0.06238 0.02592 =

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			0.03453	0.00048	0.01951	-0.00305		
AFIX	43							
H4	2		0.058867	0.242347	0.275217	11.00000	-1.20000	
AFIX	0							
C5	1		0.144841	0.562909	0.312668	11.00000	0.06205	0.04274 =
			0.03372	0.00060	0.02029	0.00492		
AFIX	43							
H5	2		0.163338	0.602651	0.269537	11.00000	-1.20000	
AFIX	0							
C6	1		0.180145	0.711742	0.372202	11.00000	0.05285	0.04255 =
			0.04515	0.00448	0.02155	-0.00748		
AFIX	43							
H6	2		0.226110	0.843243	0.369633	11.00000	-1.20000	
AFIX	0							
C7	1		0.151817	0.679486	0.436522	11.00000	0.05208	0.03954 =
			0.02976	-0.00880	0.01705	-0.00315		
AFIX	43							
H7	2		0.174403	0.791723	0.476370	11.00000	-1.20000	
AFIX	0							
C8	1		0.087602	0.469535	0.438204	11.00000	0.04569	0.02151 =
			0.03069	-0.00008	0.01544	0.00116		
C9	1		0.041328	0.401359	0.498855	11.00000	0.04950	0.02772 =
			0.02598	-0.00159	0.00939	-0.00216		
C10	1		0.178491	0.324245	0.586451	11.00000	0.03787	0.04416 =
			0.03226	-0.00144	0.00916	-0.00713		
C11	1		0.225998	0.437344	0.656181	11.00000	0.05296	0.04656 =
			0.03961	-0.01005	0.00721	0.00985		
AFIX	23							
H11A	2		0.181588	0.453970	0.687478	11.00000	-1.20000	
H11B	2		0.248190	0.624811	0.648716	11.00000	-1.20000	
AFIX	0							
C12	1		0.305532	0.264225	0.693309	11.00000	0.04635	0.06014 =
			0.03299	0.00110	0.00588	0.00665		
AFIX	23							
H12A	2		0.284485	0.074221	0.699391	11.00000	-1.20000	
H12B	2		0.352004	0.255436	0.663617	11.00000	-1.20000	
AFIX	0							
C13	1		0.347560	0.382603	0.764832	11.00000	0.04751	0.06581 =
			0.04797	-0.01173	-0.00296	0.00599		
AFIX	23							
H13A	2		0.371222	0.568421	0.757614	11.00000	-1.20000	
H13B	2		0.299138	0.405110	0.792347	11.00000	-1.20000	
AFIX	0							
PART	1	11						

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C14  1  0.422326  0.218133  0.807857  11.00000  0.06041  0.06381 =
      0.03855 -0.00302  0.00578  0.00809
AFIX 23
H14A 2  0.470973  0.196715  0.780516  11.00000 -1.20000
H14B 2  0.398813  0.031915  0.814852  11.00000 -1.20000
AFIX  0
C15  1  0.463483  0.335665  0.879084  11.00000  0.07090  0.06993 =
      0.04494 -0.01400  0.00586  0.00843
AFIX 23
H15A 2  0.492047  0.514363  0.871970  11.00000 -1.20000
H15B 2  0.414142  0.372797  0.904888  11.00000 -1.20000
AFIX  0
C16  1  0.531278  0.161341  0.923340  11.00000  0.06620  0.08320 =
      0.04257  0.00466  0.00738  0.00925
AFIX 43
H16  2  0.511348 -0.017665  0.932993  11.00000 -1.20000
AFIX  0
C17  1  0.613918  0.217933  0.950800  11.00000  0.06395  0.12148 =
      0.06583  0.02449  0.00311 -0.01631
AFIX 93
H17A 2  0.638207  0.393440  0.943232  11.00000 -1.20000
H17B 2  0.650877  0.084368  0.978719  11.00000 -1.20000
AFIX  0
HKLF 4

```

REM PBIT_Oct2_D_2_0m_a.res in P2(1)/c

REM wR2 = 0.4046, GooF = S = 1.145, Restrained GooF = 1.145 for all data

REM R1 = 0.1447 for 1297 Fo > 4sig(Fo) and 0.2054 for all 2360 data

REM 181 parameters refined using 0 restraints

END

WGHT 0.1246 10.2917

REM Highest difference peak 0.390, deepest hole -0.445, 1-sigma level 0.104

```

Q1  1  0.5120  0.7089  0.8585  11.00000  0.05  0.39
Q2  1 -0.1672 -0.0588  0.3412  11.00000  0.05  0.39
Q3  1  0.0974  0.3226  0.6246  11.00000  0.05  0.38
Q4  1  0.0601  0.2100  0.2307  11.00000  0.05  0.36
Q5  1  0.2262  0.2420  0.7528  11.00000  0.05  0.35
Q6  1  0.1355  0.0883  0.6098  11.00000  0.05  0.34
Q7  1  0.3210  0.6196  0.6133  11.00000  0.05  0.33
Q8  1  0.0226  0.3557  0.2830  11.00000  0.05  0.33

```

Q9	1	0.3871	0.2837	0.6010	11.00000	0.05	0.31
Q10	1	0.0705	0.0995	0.3773	11.00000	0.05	0.31
Q11	1	0.2310	0.6126	0.4784	11.00000	0.05	0.31
Q12	1	0.1948	0.9050	0.6563	11.00000	0.05	0.30
Q13	1	0.3089	0.5717	0.7820	11.00000	0.05	0.29
Q14	1	0.6786	-0.1574	0.9662	11.00000	0.05	0.29
Q15	1	0.6856	0.3098	1.0509	11.00000	0.05	0.29
Q16	1	0.0075	-0.0520	0.3620	11.00000	0.05	0.29
Q17	1	0.1296	0.1934	0.5312	11.00000	0.05	0.29
Q18	1	0.3517	0.2115	0.8606	11.00000	0.05	0.29
Q19	1	0.1666	0.5220	0.5113	11.00000	0.05	0.29
Q20	1	0.1747	0.7162	0.2762	11.00000	0.05	0.29

;

_shelx_res_checksum 38984

polyME-CB (see CCDC-2088149 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C10 H8 Cl O2'
_chemical_formula_weight    195.61
```

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  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
'C' 'C' 0.0033 0.0016
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H' 'H' 0.0000 0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O' 'O' 0.0106 0.0060
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'Cl' 'Cl' 0.1484 0.1585
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

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_space_group_crystal_system monoclinic
_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
'x, y, z'
'-x, y+1/2, -z+1/2'
```

'-x, -y, -z'
'x, -y-1/2, z-1/2'

_cell_length_a	5.6290(5)
_cell_length_b	4.8714(4)
_cell_length_c	31.620(3)
_cell_angle_alpha	90
_cell_angle_beta	91.060(4)
_cell_angle_gamma	90
_cell_volume	866.89(13)
_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflms_used	3179
_cell_measurement_theta_min	3.6198
_cell_measurement_theta_max	33.1116

_exptl_crystal_description	needle
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.499
_exptl_crystal_F_000	404
_exptl_transmission_factor_min	?
_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.550
_exptl_crystal_size_mid	0.110
_exptl_crystal_size_min	0.040
_exptl_absorpt_coefficient_mu	0.398
_shelx_estimated_absorpt_T_min	0.811
_shelx_estimated_absorpt_T_max	0.984
_exptl_absorpt_correction_type	multi-scan
_exptl_absorpt_correction_T_min	0.6062
_exptl_absorpt_correction_T_max	0.7465
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK α
_diffn_source	'fine focus sealed tube X-ray source'
_diffn_source_current	'30 mA'

```

_diffn_source_voltage      '50 kV'
_diffn_radiation_monochromator 'Triumph curved graphite crystal'
_diffn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel
array detector (CPAD)
;
_diffn_measurement_method    'omega and phi scans'
_diffn_detector_area_resol_mean 7.4074
_diffn_reflns_number         9491
_diffn_reflns_av_unetI/netI    0.0843
_diffn_reflns_av_R_equivalents 0.0866
_diffn_reflns_limit_h_min     -7
_diffn_reflns_limit_h_max      8
_diffn_reflns_limit_k_min     -6
_diffn_reflns_limit_k_max      7
_diffn_reflns_limit_l_min     -45
_diffn_reflns_limit_l_max      48
_diffn_reflns_theta_min       2.577
_diffn_reflns_theta_max       33.132
_diffn_reflns_theta_full      25.242
_diffn_measured_fraction_theta_max 0.899
_diffn_measured_fraction_theta_full 0.998
_diffn_reflns_Laue_measured_fraction_max 0.899
_diffn_reflns_Laue_measured_fraction_full 0.998
_diffn_reflns_point_group_measured_fraction_max 0.899
_diffn_reflns_point_group_measured_fraction_full 0.998
_reflns_number_total          2985
_reflns_number_gt             2088
_reflns_threshold_expression   'I > 2\sigma(I)'
_reflns_Friedel_coverage      0.000
_reflns_Friedel_fraction_max  .
_reflns_Friedel_fraction_full .

```

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_reflns_special_details
;

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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.

```

;

```

```

_computing_data_collection      'Apex3 v2019.11-0 (Bruker, 2020)'
_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction       'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution    'SHELXT (Sheldrick, 2015)'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1215 (Hübschle et al., 2011)
;
_computing_molecular_graphics   ?
_computing_publication_material ?
_refine_special_details         ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type          full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'w=1/[s^2(Fo^2)+(0.0568P)^2] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary     direct
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        .
_refine_ls_number_reflns         2985
_refine_ls_number_parameters      118
_refine_ls_number_restraints      0
_refine_ls_R_factor_all          0.0853
_refine_ls_R_factor_gt           0.0440
_refine_ls_wR_factor_ref         0.1171
_refine_ls_wR_factor_gt         0.1060
_refine_ls_goodness_of_fit_ref    1.043
_refine_ls_restrained_S_all      1.043
_refine_ls_shift/su_max          0.001
_refine_ls_shift/su_mean         0.000

```

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_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
 Cl1 Cl 0.09391(7) -0.56956(8) 0.28613(2) 0.02239(11) Uani 1 1 d
 O1 O 0.8566(2) 0.5924(3) 0.44317(4) 0.0358(4) Uani 1 1 d
 O2 O 0.53292(19) 0.3713(2) 0.41879(3) 0.0176(2) Uani 1 1 d
 C1 C 0.5703(3) 0.8908(3) 0.49934(4) 0.0162(2) Uani 1 1 d
 H1 H 0.729292 0.909617 0.509547 0.019 Uiso 1 1 calc R U . . .
 C2 C 0.4928(2) 0.6151(3) 0.48260(4) 0.0144(2) Uani 1 1 d
 H2 H 0.325232 0.629151 0.471765 0.017 Uiso 1 1 calc R U . . .
 C3 C 0.6511(3) 0.5297(3) 0.44658(4) 0.0174(3) Uani 1 1 d
 C4 C 0.6666(3) 0.2685(3) 0.38389(5) 0.0207(3) Uani 1 1 d
 H4A H 0.816269 0.184029 0.394435 0.025 Uiso 1 1 calc R U . . .
 H4B H 0.707262 0.421113 0.364648 0.025 Uiso 1 1 calc R U . . .
 C5 C 0.5191(3) 0.0579(3) 0.36043(4) 0.0162(2) Uani 1 1 d
 C6 C 0.5931(3) -0.0294(3) 0.32094(5) 0.0194(3) Uani 1 1 d
 H6 H 0.734319 0.044681 0.309529 0.023 Uiso 1 1 calc R U . . .
 C7 C 0.4643(3) -0.2230(3) 0.29790(4) 0.0194(3) Uani 1 1 d
 H7 H 0.516023 -0.281985 0.270957 0.023 Uiso 1 1 calc R U . . .
 C8 C 0.2577(3) -0.3289(3) 0.31509(4) 0.0168(3) Uani 1 1 d
 C9 C 0.1828(3) -0.2479(3) 0.35451(5) 0.0199(3) Uani 1 1 d
 H9 H 0.043434 -0.325029 0.366127 0.024 Uiso 1 1 calc R U . . .
 C10 C 0.3133(3) -0.0524(3) 0.37710(5) 0.0188(3) Uani 1 1 d
 H10 H 0.261273 0.006088 0.404056 0.023 Uiso 1 1 calc R U . . .

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 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
 Cl1 0.0246(2) 0.01944(17) 0.02302(17) -0.00410(12) -0.00227(14) -0.00705(12)
 O1 0.0262(6) 0.0480(9) 0.0333(7) -0.0209(6) 0.0081(6) -0.0163(6)
 O2 0.0204(5) 0.0162(5) 0.0162(4) -0.0056(3) 0.0017(4) -0.0021(3)
 C1 0.0230(7) 0.0079(5) 0.0178(5) -0.0004(4) 0.0008(5) -0.0021(4)
 C2 0.0192(6) 0.0085(5) 0.0156(5) -0.0013(4) 0.0019(5) -0.0013(4)
 C3 0.0231(7) 0.0116(5) 0.0174(5) -0.0023(4) 0.0003(5) -0.0024(4)

C4 0.0216(7) 0.0217(7) 0.0188(6) -0.0076(5) 0.0034(6) -0.0055(5)
 C5 0.0198(6) 0.0128(5) 0.0159(5) -0.0024(4) 0.0011(5) -0.0011(4)
 C6 0.0209(7) 0.0197(6) 0.0179(5) -0.0046(5) 0.0054(6) -0.0065(5)
 C7 0.0237(7) 0.0176(6) 0.0170(6) -0.0041(5) 0.0037(6) -0.0045(5)
 C8 0.0203(6) 0.0134(6) 0.0167(5) -0.0018(4) -0.0020(5) -0.0022(4)
 C9 0.0206(7) 0.0199(7) 0.0192(6) -0.0017(5) 0.0030(6) -0.0057(5)
 C10 0.0198(7) 0.0195(6) 0.0172(6) -0.0039(5) 0.0032(5) -0.0030(5)

_geom_special_details

;

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.

;

loop_

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_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

C11 C8 1.7410(13) . ?

O1 C3 1.203(2) . ?

O2 C3 1.3370(15) . ?

O2 C4 1.4373(19) . ?

C1 C1 1.327(3) 3_676 ?

C1 C2 1.5053(19) . ?

C1 H1 0.9500 . ?

C2 C3 1.517(2) . ?

C2 C2 1.572(3) 3_666 ?

C2 H2 1.0000 . ?

C4 C5 1.5067(19) . ?

C4 H4A 0.9900 . ?

C4 H4B 0.9900 . ?

C5 C10 1.389(2) . ?

C5 C6 1.391(2) . ?

C6 C7 1.3879(19) . ?

C6 H6 0.9500 . ?

C7 C8 1.392(2) . ?

C7 H7 0.9500 . ?

C8 C9 1.381(2) . ?

C9 C10 1.3917(19) . ?

C9 H9 0.9500 . ?

C10 H10 0.9500 . ?

loop_

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_geom_angle_atom_site_label_3

_geom_angle

_geom_angle_site_symmetry_1

_geom_angle_site_symmetry_3

_geom_angle_publ_flag

C3 O2 C4 116.38(12) . . ?

C1 C1 C2 123.85(16) 3_676 . ?

C1 C1 H1 118.1 3_676 . ?

C2 C1 H1 118.1 . . ?

C1 C2 C3 109.76(12) . . ?

C1 C2 C2 112.31(13) . 3_666 ?

C3 C2 C2 107.85(14) . 3_666 ?

C1 C2 H2 109.0 . . ?

C3 C2 H2 109.0 . . ?

C2 C2 H2 109.0 3_666 . ?

O1 C3 O2 123.72(15) . . ?

O1 C3 C2 125.20(13) . . ?

O2 C3 C2 111.08(12) . . ?

O2 C4 C5 108.88(12) . . ?

O2 C4 H4A 109.9 . . ?

C5 C4 H4A 109.9 . . ?

O2 C4 H4B 109.9 . . ?

C5 C4 H4B 109.9 . . ?

H4A C4 H4B 108.3 . . ?

C10 C5 C6 119.24(13) . . ?

C10 C5 C4 122.16(13) . . ?

C6 C5 C4 118.59(13) . . ?

C7 C6 C5 121.10(14) . . ?

C7 C6 H6 119.5 . . ?

C5 C6 H6 119.5 . . ?

C6 C7 C8 118.64(14) . . ?

C6 C7 H7 120.7 . . ?

C8 C7 H7 120.7 . . ?

C9 C8 C7 121.17(13) . . ?

C9 C8 Cl1 119.98(11) . . ?

C7 C8 Cl1 118.85(11) . . ?

C8 C9 C10 119.47(14) . . ?

C8 C9 H9 120.3 . . ?
 C10 C9 H9 120.3 . . ?
 C5 C10 C9 120.36(14) . . ?
 C5 C10 H10 119.8 . . ?
 C9 C10 H10 119.8 . . ?

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 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
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 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
 C1 C1 C2 C3 122.0(2) 3_676 . . . ?
 C1 C1 C2 C2 -118.0(2) 3_676 . . 3_666 ?
 C4 O2 C3 O1 1.6(2) ?
 C4 O2 C3 C2 -177.80(12) ?
 C1 C2 C3 O1 30.4(2) ?
 C2 C2 C3 O1 -92.2(2) 3_666 . . . ?
 C1 C2 C3 O2 -150.14(11) ?
 C2 C2 C3 O2 87.21(15) 3_666 . . . ?
 C3 O2 C4 C5 170.32(12) ?
 O2 C4 C5 C10 -12.8(2) ?
 O2 C4 C5 C6 167.55(13) ?
 C10 C5 C6 C7 0.4(2) ?
 C4 C5 C6 C7 180.00(14) ?
 C5 C6 C7 C8 0.1(2) ?
 C6 C7 C8 C9 -1.0(2) ?
 C6 C7 C8 C11 179.43(12) ?
 C7 C8 C9 C10 1.4(2) ?
 C11 C8 C9 C10 -179.03(11) ?
 C6 C5 C10 C9 0.0(2) ?
 C4 C5 C10 C9 -179.58(14) ?
 C8 C9 C10 C5 -0.9(2) ?

_refine_diff_density_max 0.594
 _refine_diff_density_min -0.316
 _refine_diff_density_rms 0.093

_shelx_res_file

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TITL Poly_MCB_0m in P2(1)/c
  Poly_MCB_0m.res
  created by SHELXL-2018/3 at 18:35:41 on 04-Mar-2021
CELL 0.71073  5.62900  4.87140  31.61950  90.0000  91.0601  90.0000
ZERR  4.00  0.00050  0.00040  0.00250  0.0000  0.0043  0.0000
LATT 1
SYMM -X, 0.5+Y, 0.5-Z
SFAC C H O CL
UNIT 40 32 8 4
LIST 6 ! automatically inserted. Change 6 to 4 for CHECKCIF!!
TEMP -123.150
SIZE 0.04 0.11 0.55

L.S. 4
BOND $H
ACTA
CONF
FMAP 2
PLAN 20
WGHT 0.056800
FVAR 0.74583
CL1  4  0.093912 -0.569562  0.286134  11.00000  0.02463  0.01944 =
      0.02302 -0.00410 -0.00227 -0.00705
O1  3  0.856566  0.592374  0.443167  11.00000  0.02624  0.04801 =
      0.03330 -0.02090  0.00813 -0.01628
O2  3  0.532917  0.371330  0.418792  11.00000  0.02035  0.01620 =
      0.01619 -0.00562  0.00165 -0.00213
C1  1  0.570340  0.890804  0.499336  11.00000  0.02298  0.00795 =
      0.01779 -0.00044  0.00083 -0.00208
AFIX 43
H1  2  0.729292  0.909617  0.509547  11.00000 -1.20000
AFIX 0
C2  1  0.492811  0.615078  0.482603  11.00000  0.01916  0.00853 =
      0.01556 -0.00134  0.00193 -0.00133
AFIX 13
H2  2  0.325232  0.629151  0.471765  11.00000 -1.20000
AFIX 0
C3  1  0.651096  0.529651  0.446575  11.00000  0.02312  0.01156 =
      0.01739 -0.00229  0.00026 -0.00240
C4  1  0.666598  0.268529  0.383887  11.00000  0.02164  0.02171 =
      0.01883 -0.00763  0.00337 -0.00552
AFIX 23
H4A 2  0.816269  0.184029  0.394435  11.00000 -1.20000

```

```

H4B  2   0.707262   0.421113   0.364648   11.00000   -1.20000
AFIX  0
C5   1   0.519058   0.057860   0.360431   11.00000   0.01980   0.01277 =
      0.01592  -0.00239   0.00107  -0.00111
C6   1   0.593147  -0.029423   0.320936   11.00000   0.02086   0.01973 =
      0.01789  -0.00463   0.00544  -0.00645
AFIX  43
H6   2   0.734319   0.044681   0.309529   11.00000   -1.20000
AFIX  0
C7   1   0.464286  -0.223002   0.297903   11.00000   0.02371   0.01756 =
      0.01699  -0.00411   0.00367  -0.00451
AFIX  43
H7   2   0.516023  -0.281985   0.270957   11.00000   -1.20000
AFIX  0
C8   1   0.257726  -0.328943   0.315089   11.00000   0.02032   0.01344 =
      0.01670  -0.00177  -0.00200  -0.00217
C9   1   0.182849  -0.247907   0.354514   11.00000   0.02059   0.01995 =
      0.01920  -0.00173   0.00303  -0.00568
AFIX  43
H9   2   0.043434  -0.325029   0.366127   11.00000   -1.20000
AFIX  0
C10  1   0.313251  -0.052360   0.377097   11.00000   0.01977   0.01955 =
      0.01720  -0.00394   0.00322  -0.00303
AFIX  43
H10  2   0.261273   0.006088   0.404056   11.00000   -1.20000
AFIX  0
HKLF  4

```

REM Poly_MCB_0m in P2(1)/c

REM wR2 = 0.1171, GooF = S = 1.043, Restrained GooF = 1.043 for all data

REM R1 = 0.0440 for 2088 Fo > 4sig(Fo) and 0.0853 for all 2985 data

REM 118 parameters refined using 0 restraints

END

```

WGHT      0.0569      0.0000

```

REM Highest difference peak 0.594, deepest hole -0.316, 1-sigma level 0.093

```

Q1   1   0.5386  0.0618  0.3380  11.00000  0.05   0.59
Q2   1   0.3935 -0.3224  0.3071  11.00000  0.05   0.58
Q3   1   0.3412 -0.2870  0.3023  11.00000  0.05   0.57
Q4   1   0.2304 -0.3075  0.3351  11.00000  0.05   0.56
Q5   1   0.7470  0.6284  0.4369  11.00000  0.05   0.53

```

Q6	1	0.5397	0.7474	0.4936	11.00000	0.05	0.52
Q7	1	0.4019	0.0419	0.3712	11.00000	0.05	0.51
Q8	1	0.5000	0.5000	0.5000	10.50000	0.05	0.49
Q9	1	0.5586	0.5888	0.4631	11.00000	0.05	0.49
Q10	1	0.0626	-0.6687	0.3005	11.00000	0.05	0.48
Q11	1	0.4398	-0.0251	0.3663	11.00000	0.05	0.47
Q12	1	0.1334	-0.4497	0.2961	11.00000	0.05	0.46
Q13	1	0.2450	-0.1325	0.3665	11.00000	0.05	0.44
Q14	1	0.4630	0.9587	0.5007	11.00000	0.05	0.43
Q15	1	0.5016	-0.1016	0.3101	11.00000	0.05	0.43
Q16	1	0.0735	-0.5703	0.3077	11.00000	0.05	0.43
Q17	1	0.5638	-0.1405	0.3121	11.00000	0.05	0.43
Q18	1	0.5938	0.1836	0.3771	11.00000	0.05	0.41
Q19	1	0.5656	0.3746	0.3991	11.00000	0.05	0.37
Q20	1	0.5394	0.1558	0.3653	11.00000	0.05	0.37

;

_shelx_res_checksum 75261

polyME-OMeB (see CCDC-2088151 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum       'C22 H22 O6'
_chemical_formula_weight    382.39
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0033 0.0016
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0106 0.0060
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

```
_space_group_crystal_system monoclinic
_space_group_IT_number      15
_space_group_name_H-M_alt   'C 2/c'
_space_group_name_Hall      '-C 2yc'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, y, -z+1/2'
  'x+1/2, y+1/2, z'
  '-x+1/2, y+1/2, -z+1/2'
```

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

_cell_length_a	34.809(2)
_cell_length_b	5.7070(3)
_cell_length_c	9.4249(5)
_cell_angle_alpha	90
_cell_angle_beta	98.424(3)
_cell_angle_gamma	90
_cell_volume	1852.10(18)
_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflns_used	7071
_cell_measurement_theta_min	2.3668
_cell_measurement_theta_max	33.0731

_exptl_crystal_description	plate
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.371
_exptl_crystal_F_000	808
_exptl_transmission_factor_min	?
_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.410
_exptl_crystal_size_mid	0.210
_exptl_crystal_size_min	0.020
_exptl_absorpt_coefficient_mu	0.100
_shelx_estimated_absorpt_T_min	0.960
_shelx_estimated_absorpt_T_max	0.998
_exptl_absorpt_correction_type	multi-scan
_exptl_absorpt_correction_T_min	0.6257
_exptl_absorpt_correction_T_max	0.7465
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
 J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	0.71073
_diffn_radiation_type	MoK α

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_diffrn_source          'fine focus sealed tube X-ray source'
_diffrn_source_current   '30 mA'
_diffrn_source_voltage   '50 kV'
_diffrn_radiation_monochromator 'Triumph curved graphite crystal'
_diffrn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel
array detector (CPAD)
;
_diffrn_measurement_method 'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number 14999
_diffrn_reflns_av_unetI/netI 0.0293
_diffrn_reflns_av_R_equivalents 0.0421
_diffrn_reflns_limit_h_min -47
_diffrn_reflns_limit_h_max 53
_diffrn_reflns_limit_k_min -8
_diffrn_reflns_limit_k_max 8
_diffrn_reflns_limit_l_min -11
_diffrn_reflns_limit_l_max 14
_diffrn_reflns_theta_min 3.550
_diffrn_reflns_theta_max 33.183
_diffrn_reflns_theta_full 25.242
_diffrn_measured_fraction_theta_max 0.953
_diffrn_measured_fraction_theta_full 0.995
_diffrn_reflns_Laue_measured_fraction_max 0.953
_diffrn_reflns_Laue_measured_fraction_full 0.995
_diffrn_reflns_point_group_measured_fraction_max 0.953
_diffrn_reflns_point_group_measured_fraction_full 0.995
_reflns_number_total 3375
_reflns_number_gt 2762
_reflns_threshold_expression 'I > 2\sigma(I)'
_reflns_Friedel_coverage 0.000
_reflns_Friedel_fraction_max .
_reflns_Friedel_fraction_full .

```

```
_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.

;

_computing_data_collection 'Apex3 v2019.11-0 (Bruker, 2020)'

_computing_cell_refinement 'SAINT V8.40B (Bruker, 2020)'

_computing_data_reduction 'SAINT V8.40B (Bruker, 2020)'

_computing_structure_solution 'SHELXT (Sheldrick, 2015)'

_computing_structure_refinement

;

SHELXL-2018/3 (Sheldrick, 2015, 2018),

SHELXLE Rev1231(H\"ubschle et al., 2011)

;

_computing_molecular_graphics ?

_computing_publication_material ?

_refine_special_details ?

_refine_ls_structure_factor_coef Fsqd

_refine_ls_matrix_type full

_refine_ls_weighting_scheme calc

_refine_ls_weighting_details

'w=1/[s^2^(Fo^2)+(0.0650P)^2+0.6437P] where P=(Fo^2+2Fc^2)/3'

_atom_sites_solution_primary dual

_atom_sites_solution_secondary difmap

_atom_sites_solution_hydrogens geom

_refine_ls_hydrogen_treatment constr

_refine_ls_extinction_method none

_refine_ls_extinction_coef .

_refine_ls_number_reflns 3375

_refine_ls_number_parameters 128

_refine_ls_number_restraints 0

_refine_ls_R_factor_all 0.0516

_refine_ls_R_factor_gt 0.0401

_refine_ls_wR_factor_ref 0.1178

_refine_ls_wR_factor_gt 0.1112

_refine_ls_goodness_of_fit_ref 1.051

_refine_ls_restrained_S_all 1.051

_refine_ls_shift/su_max 0.001

_refine_ls_shift/su_mean 0.000

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

O1 O 0.46076(3) 0.01640(14) 0.81495(9) 0.0322(2) Uani 1 1 d
 O2 O 0.42811(2) 0.25872(11) 0.65272(7) 0.01877(14) Uani 1 1 d
 O3 O 0.28363(2) 0.30945(13) 0.15337(7) 0.02255(16) Uani 1 1 d
 C1 C 0.49949(2) 0.40679(14) 0.95737(8) 0.01508(16) Uani 1 1 d
 H1 H 0.509250 0.262082 0.997442 0.018 Uiso 1 1 calc R U . . .
 C2 C 0.48345(2) 0.41544(14) 0.79930(8) 0.01435(15) Uani 1 1 d
 H2 H 0.467914 0.562451 0.780270 0.017 Uiso 1 1 calc R U . . .
 C3 C 0.45673(3) 0.20754(15) 0.75989(9) 0.01619(16) Uani 1 1 d
 C4 C 0.40517(3) 0.05942(16) 0.59594(10) 0.02164(18) Uani 1 1 d
 H4A H 0.393059 -0.015540 0.673132 0.026 Uiso 1 1 calc R U . . .
 H4B H 0.422050 -0.057528 0.557977 0.026 Uiso 1 1 calc R U . . .
 C5 C 0.37419(3) 0.14035(15) 0.47800(9) 0.01720(16) Uani 1 1 d
 C6 C 0.37714(3) 0.34202(16) 0.39768(9) 0.01926(17) Uani 1 1 d
 H6 H 0.399545 0.438181 0.418081 0.023 Uiso 1 1 calc R U . . .
 C7 C 0.34755(3) 0.40581(16) 0.28695(9) 0.01900(17) Uani 1 1 d
 H7 H 0.349897 0.544159 0.232819 0.023 Uiso 1 1 calc R U . . .
 C8 C 0.31465(3) 0.26456(16) 0.25690(9) 0.01701(16) Uani 1 1 d
 C9 C 0.31170(3) 0.05946(16) 0.33556(9) 0.01898(17) Uani 1 1 d
 H9 H 0.289570 -0.038703 0.314157 0.023 Uiso 1 1 calc R U . . .
 C10 C 0.34117(3) -0.00031(16) 0.44490(9) 0.01918(17) Uani 1 1 d
 H10 H 0.338901 -0.139427 0.498404 0.023 Uiso 1 1 calc R U . . .
 C11 C 0.28508(3) 0.51778(19) 0.07015(11) 0.0252(2) Uani 1 1 d
 H11A H 0.309074 0.519005 0.027218 0.038 Uiso 1 1 calc R U . . .
 H11B H 0.284507 0.655240 0.132124 0.038 Uiso 1 1 calc R U . . .
 H11C H 0.262653 0.521935 -0.005957 0.038 Uiso 1 1 calc R U . . .

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 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12

O1 0.0392(4) 0.0209(4) 0.0307(4) 0.0118(3) -0.0142(3) -0.0075(3)
 O2 0.0205(3) 0.0175(3) 0.0160(3) 0.0014(2) -0.0052(2) -0.0016(2)
 O3 0.0198(3) 0.0255(3) 0.0202(3) 0.0032(2) -0.0041(2) -0.0009(2)
 C1 0.0201(4) 0.0161(4) 0.0083(3) 0.0009(2) -0.0004(3) 0.0020(3)
 C2 0.0200(4) 0.0139(3) 0.0084(3) 0.0004(2) -0.0006(2) 0.0011(3)
 C3 0.0199(4) 0.0172(4) 0.0107(3) 0.0014(2) -0.0006(3) -0.0002(3)
 C4 0.0240(4) 0.0184(4) 0.0200(4) 0.0026(3) -0.0054(3) -0.0046(3)
 C5 0.0190(4) 0.0179(4) 0.0140(3) 0.0002(3) -0.0002(3) -0.0022(3)
 C6 0.0191(4) 0.0200(4) 0.0177(4) 0.0019(3) -0.0007(3) -0.0044(3)
 C7 0.0204(4) 0.0190(4) 0.0168(3) 0.0032(3) 0.0002(3) -0.0024(3)
 C8 0.0174(4) 0.0200(4) 0.0131(3) -0.0011(3) 0.0005(3) 0.0003(3)
 C9 0.0195(4) 0.0199(4) 0.0168(3) -0.0010(3) 0.0002(3) -0.0045(3)
 C10 0.0224(4) 0.0178(4) 0.0165(4) 0.0019(3) 0.0000(3) -0.0048(3)
 C11 0.0265(5) 0.0254(5) 0.0219(4) 0.0044(3) -0.0026(3) 0.0040(4)

_geom_special_details

;

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.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

O1 C3 1.2074(11) . ?

O2 C3 1.3429(10) . ?

O2 C4 1.4464(11) . ?

O3 C8 1.3694(10) . ?

O3 C11 1.4293(12) . ?

C1 C1 1.3306(16) 5_667 ?

C1 C2 1.5133(11) . ?

C1 H1 0.9500 . ?

C2 C3 1.5196(12) . ?

C2 C2 1.5829(16) 2_656 ?

C2 H2 1.0000 . ?

C4 C5 1.5036(12) . ?

C4 H4A 0.9900 . ?

C4 H4B 0.9900 . ?
 C5 C6 1.3897(12) . ?
 C5 C10 1.3992(12) . ?
 C6 C7 1.4027(12) . ?
 C6 H6 0.9500 . ?
 C7 C8 1.3952(12) . ?
 C7 H7 0.9500 . ?
 C8 C9 1.3974(12) . ?
 C9 C10 1.3858(12) . ?
 C9 H9 0.9500 . ?
 C10 H10 0.9500 . ?
 C11 H11A 0.9800 . ?
 C11 H11B 0.9800 . ?
 C11 H11C 0.9800 . ?

loop_
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 _geom_angle_atom_site_label_3
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 _geom_angle_site_symmetry_3
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 C3 O2 C4 114.57(7) . . ?
 C8 O3 C11 117.32(7) . . ?
 C1 C1 C2 122.81(9) 5_667 . ?
 C1 C1 H1 118.6 5_667 . ?
 C2 C1 H1 118.6 . . ?
 C1 C2 C3 109.85(7) . . ?
 C1 C2 C2 112.49(8) . 2_656 ?
 C3 C2 C2 109.19(5) . 2_656 ?
 C1 C2 H2 108.4 . . ?
 C3 C2 H2 108.4 . . ?
 C2 C2 H2 108.4 2_656 . ?
 O1 C3 O2 123.12(8) . . ?
 O1 C3 C2 124.89(8) . . ?
 O2 C3 C2 111.97(7) . . ?
 O2 C4 C5 109.22(7) . . ?
 O2 C4 H4A 109.8 . . ?
 C5 C4 H4A 109.8 . . ?
 O2 C4 H4B 109.8 . . ?
 C5 C4 H4B 109.8 . . ?
 H4A C4 H4B 108.3 . . ?
 C6 C5 C10 118.53(8) . . ?

C6 C5 C4 123.70(8) . . ?
 C10 C5 C4 117.74(8) . . ?
 C5 C6 C7 121.03(8) . . ?
 C5 C6 H6 119.5 . . ?
 C7 C6 H6 119.5 . . ?
 C8 C7 C6 119.50(8) . . ?
 C8 C7 H7 120.2 . . ?
 C6 C7 H7 120.2 . . ?
 O3 C8 C7 125.08(8) . . ?
 O3 C8 C9 115.04(8) . . ?
 C7 C8 C9 119.88(8) . . ?
 C10 C9 C8 119.79(8) . . ?
 C10 C9 H9 120.1 . . ?
 C8 C9 H9 120.1 . . ?
 C9 C10 C5 121.26(8) . . ?
 C9 C10 H10 119.4 . . ?
 C5 C10 H10 119.4 . . ?
 O3 C11 H11A 109.5 . . ?
 O3 C11 H11B 109.5 . . ?
 H11A C11 H11B 109.5 . . ?
 O3 C11 H11C 109.5 . . ?
 H11A C11 H11C 109.5 . . ?
 H11B C11 H11C 109.5 . . ?

loop_

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 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
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 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
 C1 C1 C2 C3 -134.00(11) 5_667 . . . ?
 C1 C1 C2 C2 104.16(11) 5_667 . . 2_656 ?
 C4 O2 C3 O1 -7.26(13) ?
 C4 O2 C3 C2 171.27(7) ?
 C1 C2 C3 O1 -32.63(12) ?
 C2 C2 C3 O1 91.16(11) 2_656 . . . ?
 C1 C2 C3 O2 148.87(7) ?
 C2 C2 C3 O2 -87.34(9) 2_656 . . . ?
 C3 O2 C4 C5 179.66(7) ?

O2 C4 C5 C6 24.79(13) ?
 O2 C4 C5 C10 -157.34(8) ?
 C10 C5 C6 C7 0.82(14) ?
 C4 C5 C6 C7 178.68(9) ?
 C5 C6 C7 C8 -0.04(14) ?
 C11 O3 C8 C7 -0.24(13) ?
 C11 O3 C8 C9 179.73(8) ?
 C6 C7 C8 O3 178.98(8) ?
 C6 C7 C8 C9 -0.99(14) ?
 O3 C8 C9 C10 -178.75(8) ?
 C7 C8 C9 C10 1.22(14) ?
 C8 C9 C10 C5 -0.43(14) ?
 C6 C5 C10 C9 -0.59(14) ?
 C4 C5 C10 C9 -178.57(9) ?

_refine_diff_density_max 0.508
 _refine_diff_density_min -0.232
 _refine_diff_density_rms 0.051

_shelx_res_file

;

TITL PM4M_0m_a.res in C2/c

PM4M_0m.res

created by SHELXL-2018/3 at 18:40:00 on 23-Apr-2021

REM Old TITL PM4M_0m in C2/c

REM SHELXT solution in C2/c

REM R1 0.118, Rweak 0.003, Alpha 0.018, Orientation as input

REM Formula found by SHELXT: C11 O3

CELL 0.71073 34.8090 5.7070 9.4249 90.000 98.424 90.000

ZERR 4.000 0.0020 0.0003 0.0005 0.000 0.003 0.000

LATT 7

SYMM -X, Y, 1/2-Z

SFAC C H O

UNIT 88 88 24

TEMP -123.150

SIZE 0.02 0.21 0.41

L.S. 10

BOND \$H

ACTA

CONF

LIST 6

FMAP 2

PLAN 20

WGHT 0.065000 0.643700

FVAR		0.50754					
O1	3	0.460760	0.016396	0.814953	11.00000	0.03921	0.02092 =
		0.03068	0.01185	-0.01423	-0.00750		
O2	3	0.428107	0.258716	0.652719	11.00000	0.02049	0.01745 =
		0.01600	0.00143	-0.00517	-0.00160		
O3	3	0.283625	0.309454	0.153370	11.00000	0.01983	0.02555 =
		0.02017	0.00325	-0.00408	-0.00089		
C1	1	0.499490	0.406791	0.957371	11.00000	0.02011	0.01610 =
		0.00827	0.00088	-0.00043	0.00199		
AFIX	43						
H1	2	0.509250	0.262082	0.997442	11.00000	-1.20000	
AFIX	0						
C2	1	0.483447	0.415441	0.799297	11.00000	0.01996	0.01387 =
		0.00841	0.00042	-0.00061	0.00110		
AFIX	13						
H2	2	0.467914	0.562451	0.780270	11.00000	-1.20000	
AFIX	0						
C3	1	0.456731	0.207545	0.759886	11.00000	0.01985	0.01720 =
		0.01066	0.00137	-0.00059	-0.00019		
C4	1	0.405174	0.059419	0.595937	11.00000	0.02396	0.01836 =
		0.02001	0.00255	-0.00541	-0.00464		
AFIX	23						
H4A	2	0.393059	-0.015540	0.673132	11.00000	-1.20000	
H4B	2	0.422050	-0.057528	0.557977	11.00000	-1.20000	
AFIX	0						
C5	1	0.374187	0.140348	0.478004	11.00000	0.01896	0.01788 =
		0.01397	0.00020	-0.00024	-0.00223		
C6	1	0.377139	0.342020	0.397679	11.00000	0.01906	0.01998 =
		0.01772	0.00194	-0.00073	-0.00443		
AFIX	43						
H6	2	0.399545	0.438181	0.418081	11.00000	-1.20000	
AFIX	0						
C7	1	0.347551	0.405813	0.286947	11.00000	0.02042	0.01899 =
		0.01684	0.00321	0.00021	-0.00244		
AFIX	43						
H7	2	0.349897	0.544159	0.232819	11.00000	-1.20000	
AFIX	0						
C8	1	0.314649	0.264559	0.256896	11.00000	0.01739	0.01999 =
		0.01312	-0.00114	0.00052	0.00027		
C9	1	0.311702	0.059459	0.335562	11.00000	0.01949	0.01987 =
		0.01683	-0.00101	0.00020	-0.00449		
AFIX	43						
H9	2	0.289570	-0.038703	0.314157	11.00000	-1.20000	
AFIX	0						

C10 1 0.341166 -0.000307 0.444903 11.00000 0.02243 0.01780 =
 0.01646 0.00186 0.00003 -0.00484
 AFIX 43
 H10 2 0.338901 -0.139427 0.498404 11.00000 -1.20000
 AFIX 0
 C11 1 0.285079 0.517781 0.070149 11.00000 0.02649 0.02543 =
 0.02188 0.00441 -0.00259 0.00405
 AFIX 137
 H11A 2 0.309074 0.519005 0.027218 11.00000 -1.50000
 H11B 2 0.284507 0.655240 0.132124 11.00000 -1.50000
 H11C 2 0.262653 0.521935 -0.005957 11.00000 -1.50000
 AFIX 0
 HKLF 4

REM PM4M_0m_a.res in C2/c

REM wR2 = 0.1178, GooF = S = 1.051, Restrained GooF = 1.051 for all data

REM R1 = 0.0401 for 2762 Fo > 4sig(Fo) and 0.0516 for all 3375 data

REM 128 parameters refined using 0 restraints

END

WGHT 0.0650 0.6440

REM Highest difference peak 0.508, deepest hole -0.232, 1-sigma level 0.051

Q1	1	0.5000	0.5000	1.0000	10.50000	0.05	0.51
Q2	1	0.3589	0.0644	0.4570	11.00000	0.05	0.48
Q3	1	0.3311	0.3282	0.2714	11.00000	0.05	0.47
Q4	1	0.4695	0.3110	0.7797	11.00000	0.05	0.42
Q5	1	0.4921	0.4245	0.8786	11.00000	0.05	0.39
Q6	1	0.3290	0.0234	0.3827	11.00000	0.05	0.39
Q7	1	0.3129	0.1596	0.2891	11.00000	0.05	0.39
Q8	1	0.3624	0.3758	0.3373	11.00000	0.05	0.37
Q9	1	0.3764	0.2390	0.4360	11.00000	0.05	0.36
Q10	1	0.3884	0.0964	0.5309	11.00000	0.05	0.32
Q11	1	0.4524	0.1214	0.8157	11.00000	0.05	0.28
Q12	1	0.4414	0.2227	0.7011	11.00000	0.05	0.28
Q13	1	0.5000	0.4293	0.7500	10.50000	0.05	0.21
Q14	1	0.3001	0.2957	0.2114	11.00000	0.05	0.20
Q15	1	0.4149	0.1987	0.6393	11.00000	0.05	0.18
Q16	1	0.4766	0.5241	0.7595	11.00000	0.05	0.18
Q17	1	0.4170	-0.0453	0.5975	11.00000	0.05	0.16
Q18	1	0.4244	0.3518	0.6289	11.00000	0.05	0.15
Q19	1	0.4870	-0.0189	0.8325	11.00000	0.05	0.14

Q20 1 0.2635 0.2632 0.1165 11.00000 0.05 0.14
;
_shelx_res_checksum 4920

polyME-Et (see CCDC-2131649 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C5 H7 O2'
_chemical_formula_weight    99.11
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
'C' 'C' 0.0181 0.0091
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H' 'H' 0.0000 0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O' 'O' 0.0492 0.0322
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

```
_space_group_crystal_system triclinic
_space_group_IT_number      2
_space_group_name_H-M_alt   'P -1'
_space_group_name_Hall      '-P 1'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
'x, y, z'
'-x, -y, -z'
```

```
_cell_length_a              4.8801(2)
```

_cell_length_b 10.0512(5)
 _cell_length_c 11.1784(6)
 _cell_angle_alpha 68.352(3)
 _cell_angle_beta 81.965(3)
 _cell_angle_gamma 76.984(3)
 _cell_volume 495.57(4)
 _cell_formula_units_Z 4
 _cell_measurement_temperature 150(2)
 _cell_measurement_reflns_used 7501
 _cell_measurement_theta_min 4.8183
 _cell_measurement_theta_max 79.3595

_exptl_crystal_description needle
 _exptl_crystal_colour colourless
 _exptl_crystal_density_meas ?
 _exptl_crystal_density_method ?
 _exptl_crystal_density_diffn 1.328
 _exptl_crystal_F_000 212
 _exptl_transmission_factor_min ?
 _exptl_transmission_factor_max ?
 _exptl_crystal_size_max 0.310
 _exptl_crystal_size_mid 0.080
 _exptl_crystal_size_min 0.050
 _exptl_absorpt_coefficient_mu 0.857
 _shelx_estimated_absorpt_T_min 0.777
 _shelx_estimated_absorpt_T_max 0.958
 _exptl_absorpt_correction_type 'multi-scan'
 _exptl_absorpt_correction_T_min 0.563256
 _exptl_absorpt_correction_T_max 0.754261
 _exptl_absorpt_process_details

;

TWINABS 2012/1: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D. (2015). J. Appl. Cryst. 48 3-10.

;

_exptl_absorpt_special_details ?
 _diffn_ambient_temperature 150(2)
 _diffn_radiation_wavelength 1.54178
 _diffn_radiation_type CuK\alpha
 _diffn_source 'I-mu-S microsource X-ray tube'
 _diffn_source_current '1.0 mA'
 _diffn_source_voltage '50 kV'
 _diffn_radiation_monochromator 'laterally graded multilayer (Goebel) mirror'
 _diffn_measurement_device_type

;

Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and photon counting pixel array detector

;

_diffn_measurement_method 'omega and phi scans'
_diffn_detector_area_resol_mean 7.4074

_diffn_reflns_number 14314
_diffn_reflns_av_unetI/netI 0.0340
_diffn_reflns_av_R_equivalents 0.0511
_diffn_reflns_limit_h_min -5
_diffn_reflns_limit_h_max 6
_diffn_reflns_limit_k_min -11
_diffn_reflns_limit_k_max 12
_diffn_reflns_limit_l_min 0
_diffn_reflns_limit_l_max 14
_diffn_reflns_theta_min 4.263
_diffn_reflns_theta_max 79.591
_diffn_reflns_theta_full 67.679
_diffn_measured_fraction_theta_max 0.971
_diffn_measured_fraction_theta_full 0.995
_diffn_reflns_Laue_measured_fraction_max 0.971
_diffn_reflns_Laue_measured_fraction_full 0.995
_diffn_reflns_point_group_measured_fraction_max 0.971
_diffn_reflns_point_group_measured_fraction_full 0.995
_reflns_number_total 3920
_reflns_number_gt 3426
_reflns_threshold_expression 'I > 2\sigma(I)'
_reflns_Friedel_coverage 0.000
_reflns_Friedel_fraction_max .
_reflns_Friedel_fraction_full .

_reflns_special_details

;

_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.

;

_computing_data_collection 'Apex4 v2021.4-0 (Bruker, 2021)'
_computing_cell_refinement 'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction 'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution 'SHELXT (Sheldrick, 2015)'
_computing_structure_refinement

```
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
```

```
;
_computing_molecular_graphics    ?
_computing_publication_material  ?
_refine_special_details
```

```
;
The crystal under investigation was found to be non-merohedrally
twinned. The orientation matrices for the two components were
identified using the program Cell_Now, with the two components
being related by a 180 degree rotation around the reciprocal
a-axis. The two components were integrated using Saint and
corrected for absorption using twinabs, resulting in the
following statistics:
```

```
6509 data (1733 unique) involve domain 1 only, mean I/sigma 19.4
6550 data (1731 unique) involve domain 2 only, mean I/sigma 17.4
1284 data (459 unique) involve 2 domains, mean I/sigma 33.9
```

```
The exact twin matrix identified by the integration program was
found to be:
```

```
1.00032 -0.19438 -0.05744
0.00313 -0.99988 -0.00113
0.00061  0.00122 -1.00044
```

```
The structure was solved using direct methods with only the
non-overlapping reflections of component 1. The structure was
refined using the hklf 5 routine with all reflections of component
1 (including the overlapping ones), resulting in a BASF value of
0.450(2).
```

```
The Rint value given is for all reflections and is based on
agreement between observed single and composite intensities
and those calculated from refined unique intensities and twin
fractions (TWINABS (Sheldrick, 2012)).
```

```
;
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
'w=1/[s^2(Fo^2)+(0.0802P)^2+0.1136P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary      dual
```

_atom_sites_solution_secondary	difmap
_atom_sites_solution_hydrogens	geom
_refine_ls_hydrogen_treatment	constr
_refine_ls_extinction_method	none
_refine_ls_extinction_coef	.
_refine_ls_number_reflns	3920
_refine_ls_number_parameters	130
_refine_ls_number_restraints	0
_refine_ls_R_factor_all	0.0567
_refine_ls_R_factor_gt	0.0516
_refine_ls_wR_factor_ref	0.1518
_refine_ls_wR_factor_gt	0.1463
_refine_ls_goodness_of_fit_ref	1.094
_refine_ls_restrained_S_all	1.094
_refine_ls_shift/su_max	0.000
_refine_ls_shift/su_mean	0.000

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	
O1A O	0.0222(3) 0.29717(13) 0.75537(11) 0.0303(3) Uani 1 1 d
O2A O	0.0895(3) 0.21699(14) 0.58849(12) 0.0349(3) Uani 1 1 d
C1A C	0.0100(3) 0.31430(18) 0.63204(16) 0.0265(4) Uani 1 1 d
C2A C	-0.1168(3) 0.46894(18) 0.55206(16) 0.0272(4) Uani 1 1 d
H2A H	-0.174693 0.529241 0.608482 0.033 Uiso 1 1 calc R U . . .
C3A C	-0.3711(4) 0.46735(18) 0.48939(17) 0.0295(4) Uani 1 1 d
H3A H	-0.342701 0.415933 0.431060 0.035 Uiso 1 1 calc R U . . .
C4A C	0.1482(4) 0.15258(18) 0.83889(16) 0.0311(4) Uani 1 1 d
H4AA H	0.095759 0.077672 0.813314 0.037 Uiso 1 1 calc R U . . .
H4AB H	0.076103 0.137799 0.929273 0.037 Uiso 1 1 calc R U . . .
C5A C	0.4639(4) 0.1366(2) 0.82878(18) 0.0360(4) Uani 1 1 d

H5AA H 0.533756 0.155482 0.738588 0.054 Uiso 1 1 calc R U . . .
 H5AB H 0.547924 0.037276 0.881465 0.054 Uiso 1 1 calc R U . . .
 H5AC H 0.515582 0.206485 0.859686 0.054 Uiso 1 1 calc R U . . .
 O1B O 0.4981(2) 0.29636(12) 0.25374(11) 0.0282(3) Uani 1 1 d
 O2B O 0.6299(3) 0.21582(13) 0.08751(12) 0.0336(3) Uani 1 1 d
 C1B C 0.5821(3) 0.31290(17) 0.13124(16) 0.0257(4) Uani 1 1 d
 C2B C 0.6125(3) 0.46860(17) 0.05182(15) 0.0255(4) Uani 1 1 d
 H2B H 0.578615 0.528128 0.108891 0.031 Uiso 1 1 calc R U . . .
 C3B C 0.9072(3) 0.46906(18) -0.01246(16) 0.0266(4) Uani 1 1 d
 H3B H 0.961300 0.422079 -0.074211 0.032 Uiso 1 1 calc R U . . .
 C4B C 0.4576(4) 0.15039(19) 0.33795(16) 0.0300(4) Uani 1 1 d
 H4BA H 0.480510 0.137343 0.428442 0.036 Uiso 1 1 calc R U . . .
 H4BB H 0.602031 0.076481 0.313829 0.036 Uiso 1 1 calc R U . . .
 C5B C 0.1686(4) 0.1288(2) 0.32647(18) 0.0341(4) Uani 1 1 d
 H5BA H 0.026001 0.203445 0.348372 0.051 Uiso 1 1 calc R U . . .
 H5BB H 0.141759 0.031947 0.385734 0.051 Uiso 1 1 calc R U . . .
 H5BC H 0.149685 0.136782 0.237808 0.051 Uiso 1 1 calc R U . . .

loop_

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 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12

O1A 0.0339(7) 0.0265(6) 0.0255(6) -0.0049(5) -0.0020(5) -0.0033(5)
 O2A 0.0403(7) 0.0306(7) 0.0324(7) -0.0108(5) -0.0030(5) -0.0036(5)
 C1A 0.0208(7) 0.0307(8) 0.0253(8) -0.0044(7) -0.0005(6) -0.0089(6)
 C2A 0.0225(8) 0.0272(8) 0.0287(9) -0.0049(7) -0.0029(6) -0.0062(6)
 C3A 0.0248(8) 0.0291(8) 0.0300(8) -0.0019(7) -0.0035(6) -0.0095(6)
 C4A 0.0327(9) 0.0270(8) 0.0254(8) -0.0016(7) -0.0004(6) -0.0036(7)
 C5A 0.0327(10) 0.0370(10) 0.0333(9) -0.0050(8) -0.0059(7) -0.0066(7)
 O1B 0.0283(6) 0.0270(6) 0.0254(6) -0.0050(5) 0.0004(5) -0.0061(5)
 O2B 0.0397(7) 0.0282(6) 0.0320(7) -0.0093(5) 0.0042(5) -0.0107(5)
 C1B 0.0187(7) 0.0277(8) 0.0274(8) -0.0054(6) -0.0022(6) -0.0046(6)
 C2B 0.0197(8) 0.0250(8) 0.0293(9) -0.0055(6) -0.0009(6) -0.0062(6)
 C3B 0.0194(7) 0.0250(7) 0.0307(8) -0.0035(6) -0.0025(6) -0.0050(6)
 C4B 0.0276(8) 0.0297(8) 0.0254(8) -0.0009(7) -0.0004(6) -0.0072(6)
 C5B 0.0289(9) 0.0372(9) 0.0330(9) -0.0075(7) 0.0018(7) -0.0104(7)

_geom_special_details

;

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.

;

```

loop_
  _geom_bond_atom_site_label_1
  _geom_bond_atom_site_label_2
  _geom_bond_distance
  _geom_bond_site_symmetry_2
  _geom_bond_publ_flag
O1A C1A 1.333(2) . ?
O1A C4A 1.4557(19) . ?
O2A C1A 1.213(2) . ?
C1A C2A 1.515(2) . ?
C2A C3A 1.514(2) . ?
C2A C2A 1.568(3) 2_566 ?
C2A H2A 1.0000 . ?
C3A C3A 1.315(4) 2_466 ?
C3A H3A 0.9500 . ?
C4A C5A 1.505(3) . ?
C4A H4AA 0.9900 . ?
C4A H4AB 0.9900 . ?
C5A H5AA 0.9800 . ?
C5A H5AB 0.9800 . ?
C5A H5AC 0.9800 . ?
O1B C1B 1.333(2) . ?
O1B C4B 1.4620(19) . ?
O2B C1B 1.210(2) . ?
C1B C2B 1.517(2) . ?
C2B C3B 1.515(2) . ?
C2B C2B 1.565(3) 2_665 ?
C2B H2B 1.0000 . ?
C3B C3B 1.318(3) 2_765 ?
C3B H3B 0.9500 . ?
C4B C5B 1.504(2) . ?
C4B H4BA 0.9900 . ?
C4B H4BB 0.9900 . ?
C5B H5BA 0.9800 . ?
C5B H5BB 0.9800 . ?
C5B H5BC 0.9800 . ?

```

loop_
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 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
 C1A O1A C4A 116.25(13) . . ?
 O2A C1A O1A 123.97(15) . . ?
 O2A C1A C2A 123.56(15) . . ?
 O1A C1A C2A 112.46(14) . . ?
 C3A C2A C1A 109.26(13) . . ?
 C3A C2A C2A 111.05(17) . 2_566 ?
 C1A C2A C2A 107.20(16) . 2_566 ?
 C3A C2A H2A 109.8 . . ?
 C1A C2A H2A 109.8 . . ?
 C2A C2A H2A 109.8 2_566 . ?
 C3A C3A C2A 124.1(2) 2_466 . ?
 C3A C3A H3A 117.9 2_466 . ?
 C2A C3A H3A 117.9 . . ?
 O1A C4A C5A 109.90(14) . . ?
 O1A C4A H4AA 109.7 . . ?
 C5A C4A H4AA 109.7 . . ?
 O1A C4A H4AB 109.7 . . ?
 C5A C4A H4AB 109.7 . . ?
 H4AA C4A H4AB 108.2 . . ?
 C4A C5A H5AA 109.5 . . ?
 C4A C5A H5AB 109.5 . . ?
 H5AA C5A H5AB 109.5 . . ?
 C4A C5A H5AC 109.5 . . ?
 H5AA C5A H5AC 109.5 . . ?
 H5AB C5A H5AC 109.5 . . ?
 C1B O1B C4B 116.39(13) . . ?
 O2B C1B O1B 124.44(15) . . ?
 O2B C1B C2B 123.41(15) . . ?
 O1B C1B C2B 112.15(13) . . ?
 C3B C2B C1B 109.20(13) . . ?
 C3B C2B C2B 110.55(16) . 2_665 ?
 C1B C2B C2B 107.75(15) . 2_665 ?
 C3B C2B H2B 109.8 . . ?
 C1B C2B H2B 109.8 . . ?
 C2B C2B H2B 109.8 2_665 . ?
 C3B C3B C2B 123.7(2) 2_765 . ?

C3B C3B H3B 118.1 2_765 . ?
 C2B C3B H3B 118.1 . . ?
 O1B C4B C5B 110.35(14) . . ?
 O1B C4B H4BA 109.6 . . ?
 C5B C4B H4BA 109.6 . . ?
 O1B C4B H4BB 109.6 . . ?
 C5B C4B H4BB 109.6 . . ?
 H4BA C4B H4BB 108.1 . . ?
 C4B C5B H5BA 109.5 . . ?
 C4B C5B H5BB 109.5 . . ?
 H5BA C5B H5BB 109.5 . . ?
 C4B C5B H5BC 109.5 . . ?
 H5BA C5B H5BC 109.5 . . ?
 H5BB C5B H5BC 109.5 . . ?

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
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
 C4A O1A C1A O2A 2.0(2) ?
 C4A O1A C1A C2A -178.43(13) ?
 O2A C1A C2A C3A 57.1(2) ?
 O1A C1A C2A C3A -122.41(15) ?
 O2A C1A C2A C2A -63.3(2) . . . 2_566 ?
 O1A C1A C2A C2A 117.15(18) . . . 2_566 ?
 C1A C2A C3A C3A 118.5(2) . . . 2_466 ?
 C2A C2A C3A C3A -123.5(2) 2_566 . . 2_466 ?
 C1A O1A C4A C5A 83.93(18) ?
 C4B O1B C1B O2B -1.3(2) ?
 C4B O1B C1B C2B 178.96(12) ?
 O2B C1B C2B C3B -54.7(2) ?
 O1B C1B C2B C3B 125.06(15) ?
 O2B C1B C2B C2B 65.4(2) . . . 2_665 ?
 O1B C1B C2B C2B -114.83(18) . . . 2_665 ?
 C1B C2B C3B C3B -115.9(2) . . . 2_765 ?
 C2B C2B C3B C3B 125.7(2) 2_665 . . 2_765 ?
 C1B O1B C4B C5B -84.43(18) ?

_refine_diff_density_max 0.491
 _refine_diff_density_min -0.176
 _refine_diff_density_rms 0.048

_shelx_res_file

;

TITL DME_ET_0m_a.res in P-1

DME_ET_0m_5.res

created by SHELXL-2018/3 at 18:47:55 on 12-Sep-2021

CELL 1.54178 4.8801 10.0512 11.1784 68.352 81.965 76.984

ZERR 4 0.0002 0.0005 0.0006 0.003 0.003 0.003

LATT 1

SFAC C H O

UNIT 20 28 8

LIST 6 ! automatically inserted. Change 6 to 4 for CHECKCIF!!

TEMP -123.15

SIZE 0.31 0.08 0.05

L.S. 5

FMAP 2

PLAN -20

ACTA

CONF

BOND \$H

TWST 0

WGHT 0.080200 0.113600

BASF 0.44953

FVAR 2.07463

O1A 3 0.022179 0.297174 0.755370 11.00000 0.03389 0.02653 =
 0.02553 -0.00486 -0.00195 -0.00327

O2A 3 0.089452 0.216994 0.588494 11.00000 0.04034 0.03055 =
 0.03244 -0.01084 -0.00302 -0.00363

C1A 1 0.009954 0.314296 0.632045 11.00000 0.02076 0.03068 =
 0.02527 -0.00441 -0.00049 -0.00886

C2A 1 -0.116847 0.468939 0.552063 11.00000 0.02251 0.02722 =
 0.02872 -0.00490 -0.00285 -0.00621

AFIX 13

H2A 2 -0.174693 0.529241 0.608482 11.00000 -1.20000

AFIX 0

C3A 1 -0.371123 0.467347 0.489391 11.00000 0.02475 0.02912 =
 0.03003 -0.00186 -0.00347 -0.00955

AFIX 43

H3A 2 -0.342701 0.415933 0.431060 11.00000 -1.20000

AFIX 0
 C4A 1 0.148205 0.152575 0.838890 11.00000 0.03272 0.02701 =
 0.02536 -0.00157 -0.00038 -0.00364
 AFIX 23
 H4AA 2 0.095759 0.077672 0.813314 11.00000 -1.20000
 H4AB 2 0.076103 0.137799 0.929273 11.00000 -1.20000
 AFIX 0
 C5A 1 0.463901 0.136558 0.828775 11.00000 0.03269 0.03697 =
 0.03329 -0.00502 -0.00589 -0.00660
 AFIX 137
 H5AA 2 0.533756 0.155482 0.738588 11.00000 -1.50000
 H5AB 2 0.547924 0.037276 0.881465 11.00000 -1.50000
 H5AC 2 0.515582 0.206485 0.859686 11.00000 -1.50000
 AFIX 0
 O1B 3 0.498136 0.296362 0.253740 11.00000 0.02829 0.02701 =
 0.02544 -0.00501 0.00043 -0.00608
 O2B 3 0.629911 0.215816 0.087510 11.00000 0.03966 0.02822 =
 0.03196 -0.00934 0.00421 -0.01074
 C1B 1 0.582074 0.312901 0.131237 11.00000 0.01868 0.02768 =
 0.02736 -0.00542 -0.00220 -0.00459
 C2B 1 0.612521 0.468599 0.051816 11.00000 0.01965 0.02498 =
 0.02926 -0.00552 -0.00093 -0.00624
 AFIX 13
 H2B 2 0.578615 0.528128 0.108891 11.00000 -1.20000
 AFIX 0
 C3B 1 0.907249 0.469060 -0.012462 11.00000 0.01935 0.02499 =
 0.03070 -0.00352 -0.00251 -0.00497
 AFIX 43
 H3B 2 0.961300 0.422079 -0.074211 11.00000 -1.20000
 AFIX 0
 C4B 1 0.457613 0.150391 0.337948 11.00000 0.02765 0.02974 =
 0.02544 -0.00086 -0.00044 -0.00718
 AFIX 23
 H4BA 2 0.480510 0.137343 0.428442 11.00000 -1.20000
 H4BB 2 0.602031 0.076481 0.313829 11.00000 -1.20000
 AFIX 0
 C5B 1 0.168584 0.128756 0.326465 11.00000 0.02891 0.03718 =
 0.03298 -0.00750 0.00176 -0.01044
 AFIX 137
 H5BA 2 0.026001 0.203445 0.348372 11.00000 -1.50000
 H5BB 2 0.141759 0.031947 0.385734 11.00000 -1.50000
 H5BC 2 0.149685 0.136782 0.237808 11.00000 -1.50000
 AFIX 0
 HKLF 5

REM DME_ET_0m_a.res in P-1
 REM wR2 = 0.1518, GooF = S = 1.094, Restrained GooF = 1.094 for all data
 REM R1 = 0.0516 for 3426 Fo > 4sig(Fo) and 0.0567 for all 3920 data
 REM 130 parameters refined using 0 restraints

END

WGHT 0.0802 0.1136

REM Highest difference peak 0.491, deepest hole -0.176, 1-sigma level 0.048

Q1	1	0.5723	0.4576	0.1938	11.00000	0.05	0.49
Q2	1	-0.1142	0.4444	0.6948	11.00000	0.05	0.44
Q3	1	0.6706	0.4184	-0.0217	11.00000	0.05	0.38
Q4	1	0.0841	0.2127	0.6965	11.00000	0.05	0.38
Q5	1	0.5810	0.3966	0.1217	11.00000	0.05	0.33
Q6	1	-0.0560	0.4118	0.4802	11.00000	0.05	0.28
Q7	1	-0.2415	0.4716	0.5144	11.00000	0.05	0.24
Q8	1	0.3234	0.1175	0.3415	11.00000	0.05	0.24
Q9	1	-0.0668	0.3919	0.6039	11.00000	0.05	0.23
Q10	1	0.5354	0.2193	0.1971	11.00000	0.05	0.23
Q11	1	0.7590	0.4741	0.0140	11.00000	0.05	0.22
Q12	1	1.0000	0.5000	0.0000	10.50000	0.05	0.22
Q13	1	-0.4815	0.5218	0.4965	11.00000	0.05	0.19
Q14	1	0.5000	0.5000	0.0000	10.50000	0.05	0.19
Q15	1	-0.3210	0.4451	0.4275	11.00000	0.05	0.18
Q16	1	0.3139	0.1368	0.8330	11.00000	0.05	0.18
Q17	1	0.0000	0.5000	0.5000	10.50000	0.05	0.18
Q18	1	0.3341	0.2322	0.3168	11.00000	0.05	0.16
Q19	1	0.3498	0.1926	0.8434	11.00000	0.05	0.15
Q20	1	0.0237	0.0770	0.8615	11.00000	0.05	0.15

;

_shelx_res_checksum 16713

polyME-NB (see CCDC-2131646 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C10 H8 N O4'
_chemical_formula_weight    206.17
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0181 0.0091
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'N' 'N' 0.0311 0.0180
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0492 0.0322
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

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_space_group_crystal_system monoclinic
_space_group_IT_number      14
_space_group_name_H-M_alt   'P 21/c'
_space_group_name_Hall      '-P 2ybc'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, y+1/2, -z+1/2'
```

'-x, -y, -z'

'x, -y-1/2, z-1/2'

_cell_length_a	5.9755(2)
_cell_length_b	4.87700(10)
_cell_length_c	31.3677(8)
_cell_angle_alpha	90
_cell_angle_beta	94.5608(14)
_cell_angle_gamma	90
_cell_volume	911.24(4)
_cell_formula_units_Z	4
_cell_measurement_temperature	150(2)
_cell_measurement_reflns_used	2730
_cell_measurement_theta_min	2.8264
_cell_measurement_theta_max	78.4650

_exptl_crystal_description	rod
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.503
_exptl_crystal_F_000	428
_exptl_transmission_factor_min	?
_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.210
_exptl_crystal_size_mid	0.050
_exptl_crystal_size_min	0.040
_exptl_absorpt_coefficient_mu	1.007
_shelx_estimated_absorpt_T_min	0.816
_shelx_estimated_absorpt_T_max	0.961
_exptl_absorpt_correction_type	multi-scan
_exptl_absorpt_correction_T_min	0.6184
_exptl_absorpt_correction_T_max	0.7542
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	1.54178
_diffn_radiation_type	CuK\alpha
_diffn_source	'I-mu-S microsource X-ray tube'
_diffn_source_current	'1.0 mA'

```

_diffn_source_voltage      '50 kV'
_diffn_radiation_monochromator  'laterally graded multilayer (Goebel) mirror'
_diffn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and
photon counting pixel array detector
;
_diffn_measurement_method    'omega and phi scans'
_diffn_detector_area_resol_mean  7.4074
_diffn_reflns_number          4747
_diffn_reflns_av_unetI/netI    0.0310
_diffn_reflns_av_R_equivalents  0.0293
_diffn_reflns_limit_h_min      -7
_diffn_reflns_limit_h_max      6
_diffn_reflns_limit_k_min      -5
_diffn_reflns_limit_k_max      6
_diffn_reflns_limit_l_min      -39
_diffn_reflns_limit_l_max      37
_diffn_reflns_theta_min        2.826
_diffn_reflns_theta_max        78.888
_diffn_reflns_theta_full       67.679
_diffn_measured_fraction_theta_max  0.930
_diffn_measured_fraction_theta_full  0.980
_diffn_reflns_Laue_measured_fraction_max  0.930
_diffn_reflns_Laue_measured_fraction_full  0.980
_diffn_reflns_point_group_measured_fraction_max  0.930
_diffn_reflns_point_group_measured_fraction_full  0.980
_reflns_number_total           1832
_reflns_number_gt              1652
_reflns_threshold_expression    'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

```

```

_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.

```

;

```

```

_computing_data_collection      'Apex4 v2021.4-0 (Bruker, 2021)'
_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction       'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution    'SHELXT (Sheldrick, 2015)'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
;
_computing_molecular_graphics   ?
_computing_publication_material ?
_refine_special_details         ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type          full
_refine_ls_weighting_scheme      calc
_refine_ls_weighting_details
'w=1/[s^2(Fo^2)+(0.0361P)^2+0.2768P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary     dual
_atom_sites_solution_secondary   difmap
_atom_sites_solution_hydrogens   geom
_refine_ls_hydrogen_treatment    constr
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        .
_refine_ls_number_reflns         1832
_refine_ls_number_parameters     136
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.0350
_refine_ls_R_factor_gt           0.0319
_refine_ls_wR_factor_ref         0.0854
_refine_ls_wR_factor_gt         0.0827
_refine_ls_goodness_of_fit_ref   1.055
_refine_ls_restrained_S_all      1.055
_refine_ls_shift/su_max          0.001
_refine_ls_shift/su_mean         0.000

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
 O1 O 0.81679(17) -0.0886(2) 0.44246(3) 0.0422(3) Uani 1 1 d
 O2 O 0.50400(13) 0.13432(16) 0.41850(2) 0.0219(2) Uani 1 1 d
 O3 O 0.16434(14) 1.16447(18) 0.26102(3) 0.0263(2) Uani 1 1 d
 O4 O -0.09980(14) 1.11308(18) 0.30357(3) 0.0288(2) Uani 1 1 d
 N1 N 0.08536(15) 1.05682(19) 0.29169(3) 0.0199(2) Uani 1 1 d
 C1 C 0.5659(2) -0.3919(2) 0.49914(3) 0.0232(2) Uani 1 1 d
 H1 H 0.719493 -0.411481 0.509067 0.028 Uiso 1 1 calc R U . . .
 C2 C 0.4872(2) -0.1152(2) 0.48232(3) 0.0218(3) Uani 1 1 d
 H2 H 0.325718 -0.128898 0.471385 0.026 Uiso 1 1 calc R U . . .
 C3 C 0.6246(2) -0.0272(2) 0.44596(3) 0.0229(2) Uani 1 1 d
 C4 C 0.62258(19) 0.2517(2) 0.38472(3) 0.0235(3) Uani 1 1 d
 H4A H 0.665104 0.105589 0.364985 0.028 Uiso 1 1 calc R U . . .
 H4B H 0.761463 0.342378 0.396899 0.028 Uiso 1 1 calc R U . . .
 C5 C 0.47323(18) 0.4576(2) 0.36079(3) 0.0194(2) Uani 1 1 d
 C6 C 0.54324(19) 0.5642(2) 0.32275(4) 0.0224(2) Uani 1 1 d
 H6 H 0.679471 0.501310 0.312464 0.027 Uiso 1 1 calc R U . . .
 C7 C 0.41701(19) 0.7599(2) 0.29986(3) 0.0215(2) Uani 1 1 d
 H7 H 0.464956 0.832957 0.274025 0.026 Uiso 1 1 calc R U . . .
 C8 C 0.21888(17) 0.8466(2) 0.31555(3) 0.0186(2) Uani 1 1 d
 C9 C 0.14393(18) 0.7449(2) 0.35311(4) 0.0229(3) Uani 1 1 d
 H9 H 0.007442 0.808349 0.363225 0.027 Uiso 1 1 calc R U . . .
 C10 C 0.27291(19) 0.5482(2) 0.37564(3) 0.0231(2) Uani 1 1 d
 H10 H 0.223998 0.475045 0.401383 0.028 Uiso 1 1 calc R U . . .

loop_

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 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
 O1 0.0412(5) 0.0493(6) 0.0381(5) 0.0227(4) 0.0155(4) 0.0207(5)
 O2 0.0301(4) 0.0181(4) 0.0181(4) 0.0047(3) 0.0054(3) 0.0023(3)
 O3 0.0283(4) 0.0252(4) 0.0259(4) 0.0092(3) 0.0047(3) 0.0028(3)
 O4 0.0267(4) 0.0309(5) 0.0296(4) 0.0022(3) 0.0078(3) 0.0114(3)

N1 0.0222(4) 0.0170(4) 0.0204(4) -0.0013(3) 0.0022(3) 0.0016(3)
 C1 0.0386(6) 0.0138(5) 0.0177(5) 0.0010(4) 0.0058(4) 0.0047(4)
 C2 0.0340(6) 0.0125(5) 0.0195(5) 0.0015(4) 0.0058(4) 0.0026(4)
 C3 0.0345(6) 0.0143(5) 0.0205(5) 0.0018(4) 0.0051(4) 0.0047(4)
 C4 0.0285(6) 0.0233(6) 0.0197(5) 0.0060(4) 0.0078(4) 0.0050(4)
 C5 0.0244(5) 0.0158(5) 0.0182(5) 0.0002(4) 0.0030(4) 0.0005(4)
 C6 0.0243(5) 0.0219(5) 0.0218(5) 0.0026(4) 0.0076(4) 0.0053(4)
 C7 0.0260(5) 0.0205(5) 0.0189(5) 0.0032(4) 0.0066(4) 0.0017(4)
 C8 0.0213(5) 0.0152(5) 0.0194(5) -0.0004(4) 0.0012(4) 0.0006(4)
 C9 0.0222(5) 0.0242(6) 0.0231(5) 0.0013(4) 0.0068(4) 0.0034(4)
 C10 0.0269(5) 0.0242(6) 0.0192(5) 0.0036(4) 0.0073(4) 0.0014(4)

_geom_special_details

;

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.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

O1 C3 1.2000(15) . ?

O2 C3 1.3355(13) . ?

O2 C4 1.4395(13) . ?

O3 N1 1.2233(12) . ?

O4 N1 1.2263(13) . ?

N1 C8 1.4672(13) . ?

C1 C1 1.320(2) 3_646 ?

C1 C2 1.5103(15) . ?

C1 H1 0.9500 . ?

C2 C3 1.5195(15) . ?

C2 C2 1.578(2) 3_656 ?

C2 H2 1.0000 . ?

C4 C5 1.5040(15) . ?

C4 H4A 0.9900 . ?

C4 H4B 0.9900 . ?

C5 C10 1.3907(16) . ?

C5 C6 1.3957(15) . ?
 C6 C7 1.3819(15) . ?
 C6 H6 0.9500 . ?
 C7 C8 1.3841(15) . ?
 C7 H7 0.9500 . ?
 C8 C9 1.3851(15) . ?
 C9 C10 1.3881(16) . ?
 C9 H9 0.9500 . ?
 C10 H10 0.9500 . ?

loop_

_geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
 C3 O2 C4 115.93(9) . . ?
 O3 N1 O4 123.83(9) . . ?
 O3 N1 C8 118.23(9) . . ?
 O4 N1 C8 117.94(9) . . ?
 C1 C1 C2 123.87(14) 3_646 . ?
 C1 C1 H1 118.1 3_646 . ?
 C2 C1 H1 118.1 . . ?
 C1 C2 C3 110.03(9) . . ?
 C1 C2 C2 112.37(11) . 3_656 ?
 C3 C2 C2 107.34(11) . 3_656 ?
 C1 C2 H2 109.0 . . ?
 C3 C2 H2 109.0 . . ?
 C2 C2 H2 109.0 3_656 . ?
 O1 C3 O2 124.02(10) . . ?
 O1 C3 C2 125.21(10) . . ?
 O2 C3 C2 110.76(9) . . ?
 O2 C4 C5 109.00(9) . . ?
 O2 C4 H4A 109.9 . . ?
 C5 C4 H4A 109.9 . . ?
 O2 C4 H4B 109.9 . . ?
 C5 C4 H4B 109.9 . . ?
 H4A C4 H4B 108.3 . . ?
 C10 C5 C6 119.50(10) . . ?
 C10 C5 C4 122.51(10) . . ?
 C6 C5 C4 117.97(9) . . ?
 C7 C6 C5 120.87(10) . . ?

C7 C6 H6 119.6 . . ?
 C5 C6 H6 119.6 . . ?
 C6 C7 C8 118.24(10) . . ?
 C6 C7 H7 120.9 . . ?
 C8 C7 H7 120.9 . . ?
 C7 C8 C9 122.47(10) . . ?
 C7 C8 N1 118.55(9) . . ?
 C9 C8 N1 118.98(9) . . ?
 C8 C9 C10 118.45(10) . . ?
 C8 C9 H9 120.8 . . ?
 C10 C9 H9 120.8 . . ?
 C9 C10 C5 120.47(10) . . ?
 C9 C10 H10 119.8 . . ?
 C5 C10 H10 119.8 . . ?

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 _geom_torsion_atom_site_label_2
 _geom_torsion_atom_site_label_3
 _geom_torsion_atom_site_label_4
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 _geom_torsion_site_symmetry_1
 _geom_torsion_site_symmetry_2
 _geom_torsion_site_symmetry_3
 _geom_torsion_site_symmetry_4
 _geom_torsion_publ_flag
 C1 C1 C2 C3 -123.08(15) 3_646 . . . ?
 C1 C1 C2 C2 117.38(16) 3_646 . . 3_656 ?
 C4 O2 C3 O1 -4.16(17) ?
 C4 O2 C3 C2 174.51(9) ?
 C1 C2 C3 O1 -30.38(17) ?
 C2 C2 C3 O1 92.18(16) 3_656 . . . ?
 C1 C2 C3 O2 150.98(9) ?
 C2 C2 C3 O2 -86.46(13) 3_656 . . . ?
 C3 O2 C4 C5 -171.92(9) ?
 O2 C4 C5 C10 11.08(15) ?
 O2 C4 C5 C6 -170.34(9) ?
 C10 C5 C6 C7 0.49(17) ?
 C4 C5 C6 C7 -178.14(10) ?
 C5 C6 C7 C8 -0.26(17) ?
 C6 C7 C8 C9 0.10(17) ?
 C6 C7 C8 N1 179.26(10) ?
 O3 N1 C8 C7 -6.42(15) ?
 O4 N1 C8 C7 173.14(10) ?

O3 N1 C8 C9 172.77(10) ?
 O4 N1 C8 C9 -7.67(15) ?
 C7 C8 C9 C10 -0.17(17) ?
 N1 C8 C9 C10 -179.32(10) ?
 C8 C9 C10 C5 0.40(17) ?
 C6 C5 C10 C9 -0.55(17) ?
 C4 C5 C10 C9 178.01(10) ?

_refine_diff_density_max 0.264
 _refine_diff_density_min -0.165
 _refine_diff_density_rms 0.037

_shelx_res_file

;

TITL PME_NB_0m_a.res in P2(1)/c

PME_NB_0m.res

created by SHELXL-2018/3 at 13:34:44 on 23-Sep-2021

REM Old TITL PME_NB_0m in P2(1)/c

REM SHELXT solution in P2(1)/c

REM R1 0.128, Rweak 0.001, Alpha 0.025, Orientation as input

REM Formula found by SHELXT: C11 O4

CELL 1.54178 5.9755 4.8770 31.3677 90.0000 94.5608 90.0000

ZERR 4.000 0.0002 0.0001 0.0008 0.0000 0.0014 0.0000

LATT 1

SYMM -X, 1/2+Y, 1/2-Z

SFAC C H N O

UNIT 40 32 4 16

TEMP -123.150

SIZE 0.04 0.05 0.21

L.S. 10

BOND \$H

ACTA

CONF

LIST 6

FMAP 2

PLAN 20

WGHT 0.036100 0.276800

FVAR 1.02685

O1 4 0.816786 -0.088569 0.442464 11.00000 0.04120 0.04934 =
 0.03809 0.02268 0.01546 0.02072

O2 4 0.503999 0.134323 0.418503 11.00000 0.03011 0.01810 =
 0.01809 0.00472 0.00539 0.00229

O3 4 0.164339 1.164469 0.261016 11.00000 0.02833 0.02521 =
 0.02589 0.00916 0.00469 0.00278

O4	4	-0.099803	1.113078	0.303567	11.00000	0.02673	0.03089 =
		0.02958	0.00219	0.00778	0.01140		
N1	3	0.085362	1.056819	0.291693	11.00000	0.02219	0.01703 =
		0.02044	-0.00135	0.00217	0.00156		
C1	1	0.565946	-0.391896	0.499140	11.00000	0.03857	0.01383 =
		0.01766	0.00101	0.00580	0.00471		
AFIX	43						
H1	2	0.719493	-0.411481	0.509067	11.00000	-1.20000	
AFIX	0						
C2	1	0.487152	-0.115248	0.482319	11.00000	0.03402	0.01254 =
		0.01955	0.00152	0.00575	0.00262		
AFIX	13						
H2	2	0.325718	-0.128898	0.471385	11.00000	-1.20000	
AFIX	0						
C3	1	0.624641	-0.027213	0.445963	11.00000	0.03446	0.01434 =
		0.02045	0.00180	0.00508	0.00474		
C4	1	0.622580	0.251652	0.384717	11.00000	0.02850	0.02332 =
		0.01969	0.00596	0.00778	0.00504		
AFIX	23						
H4A	2	0.665104	0.105589	0.364985	11.00000	-1.20000	
H4B	2	0.761463	0.342378	0.396899	11.00000	-1.20000	
AFIX	0						
C5	1	0.473230	0.457596	0.360786	11.00000	0.02443	0.01576 =
		0.01815	0.00017	0.00299	0.00046		
C6	1	0.543240	0.564156	0.322751	11.00000	0.02429	0.02193 =
		0.02179	0.00262	0.00759	0.00531		
AFIX	43						
H6	2	0.679471	0.501310	0.312464	11.00000	-1.20000	
AFIX	0						
C7	1	0.417012	0.759872	0.299855	11.00000	0.02601	0.02048 =
		0.01890	0.00325	0.00656	0.00165		
AFIX	43						
H7	2	0.464956	0.832957	0.274025	11.00000	-1.20000	
AFIX	0						
C8	1	0.218876	0.846601	0.315553	11.00000	0.02129	0.01517 =
		0.01938	-0.00037	0.00121	0.00060		
C9	1	0.143928	0.744943	0.353115	11.00000	0.02219	0.02419 =
		0.02305	0.00127	0.00679	0.00340		
AFIX	43						
H9	2	0.007442	0.808349	0.363225	11.00000	-1.20000	
AFIX	0						
C10	1	0.272910	0.548250	0.375639	11.00000	0.02686	0.02422 =
		0.01925	0.00359	0.00733	0.00144		
AFIX	43						

H10 2 0.223998 0.475045 0.401383 11.00000 -1.20000
 AFIX 0
 HKLF 4

REM PME_NB_0m_a.res in P2(1)/c
 REM wR2 = 0.0854, GooF = S = 1.055, Restrained GooF = 1.055 for all data
 REM R1 = 0.0319 for 1652 Fo > 4sig(Fo) and 0.0350 for all 1832 data
 REM 136 parameters refined using 0 restraints

END

WGHT 0.0361 0.2768

REM Highest difference peak 0.264, deepest hole -0.165, 1-sigma level 0.037

Q1	1	0.5000	0.0000	0.5000	10.50000	0.05	0.26
Q2	1	0.5295	-0.2465	0.4913	11.00000	0.05	0.23
Q3	1	0.5343	0.3444	0.3712	11.00000	0.05	0.22
Q4	1	0.5381	-0.0865	0.4637	11.00000	0.05	0.21
Q5	1	0.3146	0.7944	0.3086	11.00000	0.05	0.19
Q6	1	0.3964	0.5197	0.3709	11.00000	0.05	0.19
Q7	1	0.1529	0.9383	0.3035	11.00000	0.05	0.17
Q8	1	0.1992	0.6136	0.3599	11.00000	0.05	0.17
Q9	1	0.4514	0.6451	0.3105	11.00000	0.05	0.17
Q10	1	0.4995	0.4941	0.3412	11.00000	0.05	0.16
Q11	1	0.5211	-0.5285	0.4822	11.00000	0.05	0.16
Q12	1	0.1636	0.7693	0.3297	11.00000	0.05	0.16
Q13	1	0.3539	0.4458	0.3609	11.00000	0.05	0.16
Q14	1	0.8074	0.0405	0.4575	11.00000	0.05	0.15
Q15	1	0.5319	0.5540	0.3461	11.00000	0.05	0.14
Q16	1	0.4857	0.7073	0.3184	11.00000	0.05	0.14
Q17	1	0.8007	-0.3528	0.5111	11.00000	0.05	0.12
Q18	1	0.3890	-0.1053	0.4787	11.00000	0.05	0.11
Q19	1	-0.0928	1.0749	0.3399	11.00000	0.05	0.11
Q20	1	0.7384	0.1776	0.3739	11.00000	0.05	0.11

;

_shelx_res_checksum 68678

polyQDM-Me (see CCDC-2088150 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C16 H16 O8'
_chemical_formula_weight    336.29
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```
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  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
'C' 'C'  0.0033  0.0016
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H' 'H'  0.0000  0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'O' 'O'  0.0106  0.0060
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

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_space_group_crystal_system  monoclinic
_space_group_IT_number       14
_space_group_name_H-M_alt    'P 21/n'
_space_group_name_Hall       '-P 2yn'
```

```
_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names.

They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
'x, y, z'
'-x+1/2, y+1/2, -z+1/2'
'-x, -y, -z'
'x-1/2, -y-1/2, z-1/2'
```

_cell_length_a 8.7239(12)
 _cell_length_b 6.8134(9)
 _cell_length_c 12.4576(17)
 _cell_angle_alpha 90
 _cell_angle_beta 101.797(8)
 _cell_angle_gamma 90
 _cell_volume 724.83(17)
 _cell_formula_units_Z 2
 _cell_measurement_temperature 150(2)
 _cell_measurement_reflms_used 8017
 _cell_measurement_theta_min 2.6156
 _cell_measurement_theta_max 32.9104

_exptl_crystal_description block
 _exptl_crystal_colour 'pale brown'
 _exptl_crystal_density_meas ?
 _exptl_crystal_density_method ?
 _exptl_crystal_density_diffn 1.541
 _exptl_crystal_F_000 352
 _exptl_transmission_factor_min ?
 _exptl_transmission_factor_max ?
 _exptl_crystal_size_max 0.450
 _exptl_crystal_size_mid 0.280
 _exptl_crystal_size_min 0.230
 _exptl_absorpt_coefficient_mu 0.125
 _shelx_estimated_absorpt_T_min 0.946
 _shelx_estimated_absorpt_T_max 0.972
 _exptl_absorpt_correction_type multi-scan
 _exptl_absorpt_correction_T_min 0.5947
 _exptl_absorpt_correction_T_max 0.7465
 _exptl_absorpt_process_details

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
 J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details ?
 _diffn_ambient_temperature 150(2)
 _diffn_radiation_wavelength 0.71073
 _diffn_radiation_type MoK α
 _diffn_source 'fine focus sealed tube X-ray source'
 _diffn_source_current '30 mA'
 _diffn_source_voltage '50 kV'
 _diffn_radiation_monochromator 'Triumph curved graphite crystal'

```

_diffrn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonII charge-integrating pixel
array detector (CPAD)
;
_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number           10577
_diffrn_reflns_av_unetI/netI    0.0558
_diffrn_reflns_av_R_equivalents 0.0627
_diffrn_reflns_limit_h_min      -10
_diffrn_reflns_limit_h_max      11
_diffrn_reflns_limit_k_min      -9
_diffrn_reflns_limit_k_max      9
_diffrn_reflns_limit_l_min      -16
_diffrn_reflns_limit_l_max      16
_diffrn_reflns_theta_min        3.180
_diffrn_reflns_theta_max        28.281
_diffrn_reflns_theta_full       25.242
_diffrn_measured_fraction_theta_max 0.998
_diffrn_measured_fraction_theta_full 0.998
_diffrn_reflns_Laue_measured_fraction_max 0.998
_diffrn_reflns_Laue_measured_fraction_full 0.998
_diffrn_reflns_point_group_measured_fraction_max 0.998
_diffrn_reflns_point_group_measured_fraction_full 0.998
_reflns_number_total            1799
_reflns_number_gt               1463
_reflns_threshold_expression     'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

_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.
;

_computing_data_collection      'Apex3 v2019.11-0 (Bruker, 2020)'

```

```

_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction      'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution  'SHELXS (Sheldrick, 2008)'
_computing_structure_refinement

```

```

;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1231(H\ubschle et al., 2011)
;

```

```

_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details        ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type         full
_refine_ls_weighting_scheme     calc
_refine_ls_weighting_details
'w=1/[s^2^(Fo^2^)+3.4495P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary    direct
_atom_sites_solution_secondary  difmap
_atom_sites_solution_hydrogens  geom
_refine_ls_hydrogen_treatment   constr
_refine_ls_extinction_method    none
_refine_ls_extinction_coef      .
_refine_ls_number_reflns        1799
_refine_ls_number_parameters     111
_refine_ls_number_restraints     0
_refine_ls_R_factor_all          0.1099
_refine_ls_R_factor_gt          0.0951
_refine_ls_wR_factor_ref         0.2331
_refine_ls_wR_factor_gt         0.2258
_refine_ls_goodness_of_fit_ref   1.165
_refine_ls_restrained_S_all      1.165
_refine_ls_shift/su_max          0.000
_refine_ls_shift/su_mean         0.000

```

```

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  _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
O1 O 0.3090(4) -0.0993(4) 1.1701(2) 0.0342(7) Uani 1 1 d . . . . .
O2 O 0.5567(4) 0.0134(4) 1.2081(2) 0.0349(7) Uani 1 1 d . . . . .
O3 O 0.2352(4) 0.1626(4) 0.9853(3) 0.0355(7) Uani 1 1 d . . . . .
O4 O 0.1763(4) -0.1308(5) 0.9054(3) 0.0434(8) Uani 1 1 d . . . . .
C1 C 0.5753(5) -0.6041(6) 1.0916(3) 0.0302(8) Uani 1 1 d . . . . .
H1 H 0.627821 -0.673038 1.154738 0.036 Uiso 1 1 calc R U . . .
C2 C 0.5442(5) -0.4050(6) 1.0991(3) 0.0302(8) Uani 1 1 d . . . . .
H2 H 0.574372 -0.341566 1.168130 0.036 Uiso 1 1 calc R U . . .
C3 C 0.4700(5) -0.2953(5) 1.0087(3) 0.0279(8) Uani 1 1 d . . . . .
C4 C 0.4372(5) -0.0732(5) 1.0198(3) 0.0269(8) Uani 1 1 d . . . . .
C5 C 0.4439(5) -0.0423(6) 1.1434(3) 0.0297(8) Uani 1 1 d . . . . .
C6 C 0.3113(6) -0.1072(7) 1.2870(4) 0.0406(10) Uani 1 1 d . . . . .
H6A H 0.211719 -0.160520 1.298851 0.061 Uiso 1 1 calc R U . . .
H6B H 0.326291 0.025284 1.317970 0.061 Uiso 1 1 calc R U . . .
H6C H 0.397417 -0.191825 1.323097 0.061 Uiso 1 1 calc R U . . .
C7 C 0.2677(5) -0.0218(6) 0.9607(3) 0.0298(8) Uani 1 1 d . . . . .
C8 C 0.0860(5) 0.2410(7) 0.9306(4) 0.0431(11) Uani 1 1 d . . . . .
H8A H 0.065841 0.204065 0.852961 0.065 Uiso 1 1 calc R U . . .
H8B H 0.087759 0.384378 0.937039 0.065 Uiso 1 1 calc R U . . .
H8C H 0.003099 0.187863 0.964702 0.065 Uiso 1 1 calc R U . . .

loop_
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_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13
_atom_site_aniso_U_12
O1 0.0397(16) 0.0312(15) 0.0341(15) -0.0005(12) 0.0132(12) -0.0003(13)
O2 0.0434(17) 0.0272(15) 0.0337(15) -0.0017(12) 0.0069(12) -0.0054(13)
O3 0.0348(16) 0.0236(14) 0.0472(18) 0.0030(12) 0.0062(13) 0.0024(12)
O4 0.0363(17) 0.0438(19) 0.0489(19) -0.0125(15) 0.0060(14) -0.0033(15)
C1 0.038(2) 0.0237(18) 0.0296(19) 0.0021(15) 0.0089(16) -0.0011(16)
C2 0.036(2) 0.0216(18) 0.034(2) -0.0010(15) 0.0094(16) -0.0016(16)
C3 0.032(2) 0.0196(17) 0.034(2) 0.0002(14) 0.0093(16) 0.0004(15)
C4 0.0315(19) 0.0200(17) 0.0301(19) -0.0004(14) 0.0083(15) -0.0015(15)
C5 0.037(2) 0.0198(17) 0.033(2) 0.0015(15) 0.0096(16) 0.0027(15)

```

C6 0.050(3) 0.040(2) 0.037(2) 0.0022(19) 0.018(2) -0.002(2)
 C7 0.0305(19) 0.029(2) 0.0313(19) 0.0015(16) 0.0098(15) 0.0015(16)
 C8 0.038(2) 0.041(2) 0.052(3) 0.011(2) 0.012(2) 0.009(2)

_geom_special_details

;

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.

;

loop_

_geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag

O1 C5 1.344(5) . ?
 O1 C6 1.453(5) . ?
 O2 C5 1.199(5) . ?
 O3 C7 1.337(5) . ?
 O3 C8 1.443(5) . ?
 O4 C7 1.199(5) . ?
 C1 C2 1.390(5) . ?
 C1 C3 1.408(6) 3_647 ?
 C1 H1 0.9500 . ?
 C2 C3 1.396(6) . ?
 C2 H2 0.9500 . ?
 C3 C4 1.552(5) . ?
 C4 C5 1.544(5) . ?
 C4 C7 1.551(6) . ?
 C4 C4 1.630(7) 3_657 ?
 C6 H6A 0.9800 . ?
 C6 H6B 0.9800 . ?
 C6 H6C 0.9800 . ?
 C8 H8A 0.9800 . ?
 C8 H8B 0.9800 . ?
 C8 H8C 0.9800 . ?

loop_

_geom_angle_atom_site_label_1

_geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
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 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
 C5 O1 C6 115.2(3) . . ?
 C7 O3 C8 116.9(4) . . ?
 C2 C1 C3 120.8(4) . 3_647 ?
 C2 C1 H1 119.6 . . ?
 C3 C1 H1 119.6 3_647 . ?
 C1 C2 C3 122.2(4) . . ?
 C1 C2 H2 118.9 . . ?
 C3 C2 H2 118.9 . . ?
 C2 C3 C1 117.1(3) . 3_647 ?
 C2 C3 C4 120.9(4) . . ?
 C1 C3 C4 122.0(4) 3_647 . ?
 C5 C4 C7 106.2(3) . . ?
 C5 C4 C3 104.6(3) . . ?
 C7 C4 C3 110.7(3) . . ?
 C5 C4 C4 109.1(4) . 3_657 ?
 C7 C4 C4 110.7(4) . 3_657 ?
 C3 C4 C4 115.0(4) . 3_657 ?
 O2 C5 O1 124.4(4) . . ?
 O2 C5 C4 124.9(4) . . ?
 O1 C5 C4 110.6(3) . . ?
 O1 C6 H6A 109.5 . . ?
 O1 C6 H6B 109.5 . . ?
 H6A C6 H6B 109.5 . . ?
 O1 C6 H6C 109.5 . . ?
 H6A C6 H6C 109.5 . . ?
 H6B C6 H6C 109.5 . . ?
 O4 C7 O3 124.5(4) . . ?
 O4 C7 C4 126.2(4) . . ?
 O3 C7 C4 109.3(3) . . ?
 O3 C8 H8A 109.5 . . ?
 O3 C8 H8B 109.5 . . ?
 H8A C8 H8B 109.5 . . ?
 O3 C8 H8C 109.5 . . ?
 H8A C8 H8C 109.5 . . ?
 H8B C8 H8C 109.5 . . ?

 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
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
C3 C1 C2 C3 -1.2(7) 3_647 ... ?
C1 C2 C3 C1 1.1(7) ... 3_647 ?
C1 C2 C3 C4 -179.9(4) .... ?
C2 C3 C4 C5 -20.4(5) .... ?
C1 C3 C4 C5 158.6(4) 3_647 ... ?
C2 C3 C4 C7 -134.4(4) .... ?
C1 C3 C4 C7 44.5(5) 3_647 ... ?
C2 C3 C4 C4 99.3(5) ... 3_657 ?
C1 C3 C4 C4 -81.8(5) 3_647 .. 3_657 ?
C6 O1 C5 O2 -5.5(6) .... ?
C6 O1 C5 C4 170.3(3) .... ?
C7 C4 C5 O2 -146.8(4) .... ?
C3 C4 C5 O2 96.0(4) .... ?
C4 C4 C5 O2 -27.5(6) 3_657 ... ?
C7 C4 C5 O1 37.4(4) .... ?
C3 C4 C5 O1 -79.7(4) .... ?
C4 C4 C5 O1 156.8(4) 3_657 ... ?
C8 O3 C7 O4 -7.5(6) .... ?
C8 O3 C7 C4 175.3(3) .... ?
C5 C4 C7 O4 -119.2(4) .... ?
C3 C4 C7 O4 -6.2(6) .... ?
C4 C4 C7 O4 122.5(5) 3_657 ... ?
C5 C4 C7 O3 57.9(4) .... ?
C3 C4 C7 O3 170.9(3) .... ?
C4 C4 C7 O3 -60.4(5) 3_657 ... ?

```

```

_refine_diff_density_max    0.522
_refine_diff_density_min    -0.360
_refine_diff_density_rms    0.090

```

```
_shelx_res_file
```

```
;
```

```
TITL PQDM_0m in P2(1)/n
```

```
PQDM_0m.res
```

```
created by SHELXL-2018/3 at 07:31:36 on 26-Apr-2021
```

CELL 0.71073 8.72390 6.81340 12.45760 90.0000 101.7972 90.0000
 ZERR 2.00 0.00120 0.00090 0.00170 0.0000 0.0078 0.0000
 LATT 1
 SYMM 0.5-X, 0.5+Y, 0.5-Z
 SFAC C H O
 UNIT 32 32 16
 LIST 6 ! automatically inserted. Change 6 to 4 for CHECKCIF!!
 TEMP -123.150
 SIZE 0.45 0.28 0.23

OMIT -1 0 1

L.S. 4
 BOND \$H
 FMAP 2
 PLAN 20
 ACTA
 CONF
 SHEL 99 0.75

WGHT		0.000000	3.449500				
FVAR		1.20637					
O1	3	0.309017	-0.099342	1.170137	11.00000	0.03970	0.03119 =
		0.03406	-0.00050	0.01322	-0.00029		
O2	3	0.556700	0.013382	1.208094	11.00000	0.04343	0.02720 =
		0.03370	-0.00168	0.00686	-0.00537		
O3	3	0.235217	0.162563	0.985258	11.00000	0.03480	0.02365 =
		0.04723	0.00304	0.00623	0.00236		
O4	3	0.176251	-0.130849	0.905376	11.00000	0.03631	0.04384 =
		0.04886	-0.01248	0.00600	-0.00334		
C1	1	0.575332	-0.604068	1.091565	11.00000	0.03803	0.02366 =
		0.02961	0.00208	0.00889	-0.00105		
AFIX	43						
H1	2	0.627821	-0.673038	1.154738	11.00000	-1.20000	
AFIX	0						
C2	1	0.544196	-0.405035	1.099138	11.00000	0.03604	0.02158 =
		0.03382	-0.00105	0.00944	-0.00160		
AFIX	43						
H2	2	0.574372	-0.341566	1.168130	11.00000	-1.20000	
AFIX	0						
C3	1	0.470015	-0.295334	1.008655	11.00000	0.03178	0.01960 =
		0.03351	0.00025	0.00932	0.00044		
C4	1	0.437222	-0.073223	1.019760	11.00000	0.03152	0.01996 =
		0.03013	-0.00039	0.00827	-0.00152		
C5	1	0.443853	-0.042338	1.143415	11.00000	0.03710	0.01978 =

```

        0.03319   0.00153   0.00963   0.00266
C6   1   0.311297 -0.107244  1.287031  11.00000  0.04951  0.03951 =
        0.03650   0.00224   0.01765  -0.00217
AFIX 137
H6A   2   0.211719 -0.160520  1.298851  11.00000 -1.50000
H6B   2   0.326291  0.025284  1.317970  11.00000 -1.50000
H6C   2   0.397417 -0.191825  1.323097  11.00000 -1.50000
AFIX  0
C7   1   0.267665 -0.021811  0.960737  11.00000  0.03051  0.02912 =
        0.03135   0.00151   0.00980   0.00153
C8   1   0.086019  0.241005  0.930609  11.00000  0.03763  0.04075 =
        0.05196   0.01131   0.01167   0.00933
AFIX 137
H8A   2   0.065841  0.204065  0.852961  11.00000 -1.50000
H8B   2   0.087759  0.384378  0.937039  11.00000 -1.50000
H8C   2   0.003099  0.187863  0.964702  11.00000 -1.50000
AFIX  0
HKLF 4

```

REM PQDM_0m in P2(1)/n

REM wR2 = 0.2331, GooF = S = 1.165, Restrained GooF = 1.165 for all data

REM R1 = 0.0951 for 1463 Fo > 4sig(Fo) and 0.1099 for all 1799 data

REM 111 parameters refined using 0 restraints

END

WGHT 0.0000 3.4495

REM Highest difference peak 0.522, deepest hole -0.360, 1-sigma level 0.090

```

Q1   1   0.4333 -0.0595  1.0798  11.00000  0.05  0.52
Q2   1   0.5791  0.0102  1.2837  11.00000  0.05  0.47
Q3   1   0.6586 -0.3139  1.1964  11.00000  0.05  0.41
Q4   1   0.4390 -0.1839  1.0167  11.00000  0.05  0.35
Q5   1   0.5381  0.0049  1.1248  11.00000  0.05  0.34
Q6   1   0.3020 -0.0492  1.0359  11.00000  0.05  0.32
Q7   1   0.2964 -0.0626  1.0796  11.00000  0.05  0.31
Q8   1   0.6726 -0.3309  1.2810  11.00000  0.05  0.30
Q9   1   0.4922 -0.2825  1.0894  11.00000  0.05  0.30
Q10  1   0.2961 -0.0964  1.2230  11.00000  0.05  0.30
Q11  1   0.1583 -0.1324  0.8168  11.00000  0.05  0.29
Q12  1   0.1639 -0.1246  1.2569  11.00000  0.05  0.29
Q13  1   0.3250  0.1844  0.9575  11.00000  0.05  0.28
Q14  1   0.5741 -0.5071  1.0927  11.00000  0.05  0.28

```

Q15	1	0.7040	0.0247	1.2476	11.00000	0.05	0.27
Q16	1	0.2860	-0.0592	0.8407	11.00000	0.05	0.27
Q17	1	-0.0002	0.1340	0.9120	11.00000	0.05	0.26
Q18	1	-0.0381	0.1580	0.7989	11.00000	0.05	0.26
Q19	1	0.3603	0.2010	1.0164	11.00000	0.05	0.26
Q20	1	0.0707	-0.1114	0.8864	11.00000	0.05	0.25

;

_shelx_res_checksum 52378

polyQDM-TCNQ (see CCDC-2131648 for more complete data)

```
_audit_creation_method      'SHELXL-2018/3'
_shelx_SHELXL_version_number '2018/3'
_chemical_name_systematic   ?
_chemical_name_common       ?
_chemical_melting_point     ?
_chemical_formula_moiety    ?
_chemical_formula_sum
'C14 H10 N2 O4'
_chemical_formula_weight    270.24
```

```
loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C' 'C' 0.0181 0.0091
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H' 'H' 0.0000 0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'N' 'N' 0.0311 0.0180
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O' 'O' 0.0492 0.0322
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
```

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_space_group_crystal_system triclinic
_space_group_IT_number      2
_space_group_name_H-M_alt   'P -1'
_space_group_name_Hall      '-P 1'
```

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_shelx_space_group_comment
```

```
;
```

The symmetry employed for this shelxl refinement is uniquely defined by the following loop, which should always be used as a source of symmetry information in preference to the above space-group names. They are only intended as comments.

```
;
```

```
loop_
  _space_group_symop_operation_xyz
  'x, y, z'
  '-x, -y, -z'
```

_cell_length_a	7.037(3)
_cell_length_b	7.860(3)
_cell_length_c	12.150(4)
_cell_angle_alpha	80.096(13)
_cell_angle_beta	86.579(14)
_cell_angle_gamma	70.638(15)
_cell_volume	624.6(4)
_cell_formula_units_Z	2
_cell_measurement_temperature	150(2)
_cell_measurement_reflns_used	1830
_cell_measurement_theta_min	3.6932
_cell_measurement_theta_max	77.1408

_exptl_crystal_description	flake
_exptl_crystal_colour	colourless
_exptl_crystal_density_meas	?
_exptl_crystal_density_method	?
_exptl_crystal_density_diffn	1.437
_exptl_crystal_F_000	280
_exptl_transmission_factor_min	?
_exptl_transmission_factor_max	?
_exptl_crystal_size_max	0.180
_exptl_crystal_size_mid	0.040
_exptl_crystal_size_min	0.010
_exptl_absorpt_coefficient_mu	0.905
_shelx_estimated_absorpt_T_min	0.854
_shelx_estimated_absorpt_T_max	0.991
_exptl_absorpt_correction_type	multi-scan
_exptl_absorpt_correction_T_min	0.5815
_exptl_absorpt_correction_T_max	0.7542
_exptl_absorpt_process_details	

;

SADABS 2016/2: Krause, L., Herbst-Irmer, R., Sheldrick G.M. & Stalke D.,
J. Appl. Cryst. 48 (2015) 3-10

;

_exptl_absorpt_special_details	?
_diffn_ambient_temperature	150(2)
_diffn_radiation_wavelength	1.54178
_diffn_radiation_type	CuK\alpha
_diffn_source	'I-mu-S microsource X-ray tube'
_diffn_source_current	'1.0 mA'
_diffn_source_voltage	'50 kV'
_diffn_radiation_monochromator	'laterally graded multilayer (Goebel) mirror'

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_diffrn_measurement_device_type
;
Bruker AXS D8 Quest diffractometer with PhotonIII_C14 charge-integrating and
photon counting pixel array detector
;
_diffrn_measurement_method      'omega and phi scans'
_diffrn_detector_area_resol_mean 7.4074
_diffrn_reflns_number           5985
_diffrn_reflns_av_unetI/netI    0.0901
_diffrn_reflns_av_R_equivalents 0.0790
_diffrn_reflns_limit_h_min      -8
_diffrn_reflns_limit_h_max      7
_diffrn_reflns_limit_k_min      -9
_diffrn_reflns_limit_k_max      9
_diffrn_reflns_limit_l_min      -14
_diffrn_reflns_limit_l_max      15
_diffrn_reflns_theta_min        3.693
_diffrn_reflns_theta_max        78.890
_diffrn_reflns_theta_full       67.679
_diffrn_measured_fraction_theta_max 0.895
_diffrn_measured_fraction_theta_full 0.969
_diffrn_reflns_Laue_measured_fraction_max 0.895
_diffrn_reflns_Laue_measured_fraction_full 0.969
_diffrn_reflns_point_group_measured_fraction_max 0.895
_diffrn_reflns_point_group_measured_fraction_full 0.969
_reflns_number_total            2404
_reflns_number_gt               1505
_reflns_threshold_expression     'I > 2\sigma(I)'
_reflns_Friedel_coverage        0.000
_reflns_Friedel_fraction_max    .
_reflns_Friedel_fraction_full   .

_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.
;

_computing_data_collection      'Apex4 v2021.4-0 (Bruker, 2021)'

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_computing_cell_refinement      'SAINT V8.40B (Bruker, 2020)'
_computing_data_reduction      'SAINT V8.40B (Bruker, 2020)'
_computing_structure_solution  'SHELXT (Sheldrick, 2015)'
_computing_structure_refinement
;
SHELXL-2018/3 (Sheldrick, 2015, 2018),
SHELXLE Rev1275(H\ubschle et al., 2011)
;
_computing_molecular_graphics  ?
_computing_publication_material ?
_refine_special_details        ?
_refine_ls_structure_factor_coef Fsqd
_refine_ls_matrix_type         full
_refine_ls_weighting_scheme    calc
_refine_ls_weighting_details
'w=1/[s^2^(Fo^2^)+(0.0707P)^2^+0.0693P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary   dual
_atom_sites_solution_secondary difmap
_atom_sites_solution_hydrogens geom
_refine_ls_hydrogen_treatment constr
_refine_ls_extinction_method   none
_refine_ls_extinction_coef     .
_refine_ls_number_reflns       2404
_refine_ls_number_parameters    183
_refine_ls_number_restraints    0
_refine_ls_R_factor_all         0.0997
_refine_ls_R_factor_gt         0.0579
_refine_ls_wR_factor_ref        0.1697
_refine_ls_wR_factor_gt        0.1434
_refine_ls_goodness_of_fit_ref  1.022
_refine_ls_restrained_S_all     1.022
_refine_ls_shift/su_max         0.000
_refine_ls_shift/su_mean        0.000

loop_
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_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

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_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
O1 O 0.6655(4) 0.5671(3) 0.13403(17) 0.0436(6) Uani 1 1 d . . . . .
O2 O 0.3693(3) 0.5180(3) 0.17899(18) 0.0444(6) Uani 1 1 d . . . . .
O3 O 0.4723(4) 0.1095(3) 0.33774(19) 0.0472(6) Uani 1 1 d . . . . .
O4 O 0.6511(3) 0.1322(3) 0.17935(17) 0.0405(5) Uani 1 1 d . . . . .
N1 N 1.0782(4) 0.1554(4) 0.1249(2) 0.0477(7) Uani 1 1 d . . . . .
N2 N 0.9901(4) 0.5640(3) 0.3191(2) 0.0460(7) Uani 1 1 d . . . . .
C1 C 0.8829(5) 0.2794(3) 0.3011(2) 0.0344(7) Uani 1 1 d . . . . .
C2 C 0.9551(5) 0.1317(4) 0.4040(2) 0.0355(7) Uani 1 1 d . . . . .
C3 C 0.9906(5) 0.1775(4) 0.5047(3) 0.0377(7) Uani 1 1 d . . . . .
H3 H 0.985126 0.298929 0.507864 0.045 Uiso 1 1 calc R U . . .
C4 C 1.0336(5) 0.0477(4) 0.6000(3) 0.0373(7) Uani 1 1 d . . . . .
H4 H 1.055720 0.080575 0.668665 0.045 Uiso 1 1 calc R U . . .
C5 C 0.9857(5) 0.2120(4) 0.1992(3) 0.0376(7) Uani 1 1 d . . . . .
C6 C 0.9422(5) 0.4417(4) 0.3104(2) 0.0369(7) Uani 1 1 d . . . . .
C7 C 0.6413(5) 0.3434(3) 0.2945(2) 0.0337(6) Uani 1 1 d . . . . .
C8 C 0.5566(5) 0.4224(4) 0.4016(2) 0.0351(7) Uani 1 1 d . . . . .
C9 C 0.5341(5) 0.3147(4) 0.5009(2) 0.0364(7) Uani 1 1 d . . . . .
H9 H 0.555549 0.188342 0.502810 0.044 Uiso 1 1 calc R U . . .
C10 C 0.4801(5) 0.3910(4) 0.5982(2) 0.0377(7) Uani 1 1 d . . . . .
H10 H 0.467851 0.314758 0.666043 0.045 Uiso 1 1 calc R U . . .
C11 C 0.5640(5) 0.4896(4) 0.1917(2) 0.0355(7) Uani 1 1 d . . . . .
C12 C 0.2649(6) 0.6613(4) 0.0899(3) 0.0524(9) Uani 1 1 d . . . . .
H12A H 0.353595 0.661127 0.024512 0.079 Uiso 1 1 calc R U . . .
H12B H 0.227680 0.779960 0.115222 0.079 Uiso 1 1 calc R U . . .
H12C H 0.143116 0.639610 0.069725 0.079 Uiso 1 1 calc R U . . .
C13 C 0.5757(5) 0.1799(4) 0.2775(2) 0.0354(7) Uani 1 1 d . . . . .
C14 C 0.6192(5) -0.0263(4) 0.1484(3) 0.0446(8) Uani 1 1 d . . . . .
H14A H 0.677722 -0.044915 0.074409 0.067 Uiso 1 1 calc R U . . .
H14B H 0.474427 -0.006839 0.146553 0.067 Uiso 1 1 calc R U . . .
H14C H 0.684050 -0.134472 0.203379 0.067 Uiso 1 1 calc R U . . .

```

```

loop_
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_atom_site_aniso_U_11
_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_23
_atom_site_aniso_U_13

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_atom_site_aniso_U_12

O1 0.0522(15) 0.0370(10) 0.0420(12) -0.0050(9) 0.0026(10) -0.0164(10)
 O2 0.0442(15) 0.0403(11) 0.0417(12) 0.0010(9) -0.0024(10) -0.0081(9)
 O3 0.0551(16) 0.0465(12) 0.0468(13) -0.0087(10) 0.0080(11) -0.0263(11)
 O4 0.0515(15) 0.0361(10) 0.0390(11) -0.0134(9) 0.0051(9) -0.0181(9)
 N1 0.0444(18) 0.0478(15) 0.0485(17) -0.0121(13) 0.0051(13) -0.0107(12)
 N2 0.0510(19) 0.0404(14) 0.0499(16) -0.0085(12) 0.0033(13) -0.0191(13)
 C1 0.0396(19) 0.0274(12) 0.0358(15) -0.0073(11) 0.0032(12) -0.0100(12)
 C2 0.0325(18) 0.0325(13) 0.0401(16) -0.0059(11) -0.0008(12) -0.0086(12)
 C3 0.0419(19) 0.0300(13) 0.0417(16) -0.0057(12) -0.0039(13) -0.0118(12)
 C4 0.0417(19) 0.0330(13) 0.0380(16) -0.0093(12) -0.0014(13) -0.0112(12)
 C5 0.0387(19) 0.0351(14) 0.0390(16) -0.0082(12) 0.0056(13) -0.0118(12)
 C6 0.0404(19) 0.0322(13) 0.0374(16) -0.0065(12) 0.0033(13) -0.0110(12)
 C7 0.0382(18) 0.0282(12) 0.0343(15) -0.0079(11) 0.0044(12) -0.0095(11)
 C8 0.0389(18) 0.0293(13) 0.0359(15) -0.0068(11) 0.0032(12) -0.0093(12)
 C9 0.0439(19) 0.0307(13) 0.0351(15) -0.0095(11) 0.0055(13) -0.0118(12)
 C10 0.045(2) 0.0310(13) 0.0344(15) -0.0053(11) 0.0029(13) -0.0098(12)
 C11 0.0355(19) 0.0303(13) 0.0396(16) -0.0105(12) 0.0021(13) -0.0072(12)
 C12 0.053(2) 0.0447(17) 0.0471(19) 0.0029(15) -0.0116(16) -0.0033(15)
 C13 0.0387(18) 0.0318(13) 0.0353(15) -0.0048(11) -0.0022(13) -0.0111(12)
 C14 0.052(2) 0.0378(15) 0.0508(19) -0.0144(13) 0.0016(15) -0.0199(14)

_geom_special_details

;

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.

;

loop_

_geom_bond_atom_site_label_1

_geom_bond_atom_site_label_2

_geom_bond_distance

_geom_bond_site_symmetry_2

_geom_bond_publ_flag

O1 C11 1.205(3) . ?

O2 C11 1.327(4) . ?

O2 C12 1.452(4) . ?

O3 C13 1.199(3) . ?

O4 C13 1.338(3) . ?

O4 C14 1.449(3) . ?

N1 C5 1.146(4) . ?
 N2 C6 1.141(4) . ?
 C1 C5 1.484(4) . ?
 C1 C6 1.491(4) . ?
 C1 C2 1.535(4) . ?
 C1 C7 1.608(4) . ?
 C2 C3 1.391(4) . ?
 C2 C4 1.395(4) 2_756 ?
 C3 C4 1.380(4) . ?
 C3 H3 0.9500 . ?
 C4 H4 0.9500 . ?
 C7 C11 1.533(4) . ?
 C7 C8 1.541(4) . ?
 C7 C13 1.550(4) . ?
 C8 C9 1.383(4) . ?
 C8 C10 1.403(4) 2_666 ?
 C9 C10 1.392(4) . ?
 C9 H9 0.9500 . ?
 C10 H10 0.9500 . ?
 C12 H12A 0.9800 . ?
 C12 H12B 0.9800 . ?
 C12 H12C 0.9800 . ?
 C14 H14A 0.9800 . ?
 C14 H14B 0.9800 . ?
 C14 H14C 0.9800 . ?

loop_

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 _geom_angle_site_symmetry_3
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 C11 O2 C12 117.3(3) . . ?
 C13 O4 C14 117.3(2) . . ?
 C5 C1 C6 105.4(2) . . ?
 C5 C1 C2 110.1(2) . . ?
 C6 C1 C2 110.2(2) . . ?
 C5 C1 C7 113.6(2) . . ?
 C6 C1 C7 108.2(2) . . ?
 C2 C1 C7 109.3(2) . . ?
 C3 C2 C4 119.3(3) . 2_756 ?
 C3 C2 C1 120.7(2) . . ?

C4 C2 C1 119.6(3) 2_756 . ?
 C4 C3 C2 120.5(2) . . ?
 C4 C3 H3 119.8 . . ?
 C2 C3 H3 119.8 . . ?
 C3 C4 C2 120.2(3) . 2_756 ?
 C3 C4 H4 119.9 . . ?
 C2 C4 H4 119.9 2_756 . ?
 N1 C5 C1 174.9(3) . . ?
 N2 C6 C1 178.7(3) . . ?
 C11 C7 C8 109.8(2) . . ?
 C11 C7 C13 105.5(2) . . ?
 C8 C7 C13 114.0(2) . . ?
 C11 C7 C1 110.6(2) . . ?
 C8 C7 C1 107.5(2) . . ?
 C13 C7 C1 109.3(2) . . ?
 C9 C8 C10 118.2(3) . 2_666 ?
 C9 C8 C7 122.9(2) . . ?
 C10 C8 C7 118.6(2) 2_666 . ?
 C8 C9 C10 120.1(2) . . ?
 C8 C9 H9 119.9 . . ?
 C10 C9 H9 119.9 . . ?
 C9 C10 C8 121.7(3) . 2_666 ?
 C9 C10 H10 119.2 . . ?
 C8 C10 H10 119.2 2_666 . ?
 O1 C11 O2 126.4(3) . . ?
 O1 C11 C7 124.6(3) . . ?
 O2 C11 C7 108.9(2) . . ?
 O2 C12 H12A 109.5 . . ?
 O2 C12 H12B 109.5 . . ?
 H12A C12 H12B 109.5 . . ?
 O2 C12 H12C 109.5 . . ?
 H12A C12 H12C 109.5 . . ?
 H12B C12 H12C 109.5 . . ?
 O3 C13 O4 124.9(3) . . ?
 O3 C13 C7 127.7(3) . . ?
 O4 C13 C7 107.4(2) . . ?
 O4 C14 H14A 109.5 . . ?
 O4 C14 H14B 109.5 . . ?
 H14A C14 H14B 109.5 . . ?
 O4 C14 H14C 109.5 . . ?
 H14A C14 H14C 109.5 . . ?
 H14B C14 H14C 109.5 . . ?

loop_

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 C5 C1 C2 C3 139.8(3) ?
 C6 C1 C2 C3 24.0(4) ?
 C7 C1 C2 C3 -94.7(3) ?
 C5 C1 C2 C4 -46.5(4) . . . 2_756 ?
 C6 C1 C2 C4 -162.4(3) . . . 2_756 ?
 C7 C1 C2 C4 78.9(3) . . . 2_756 ?
 C4 C2 C3 C4 -0.9(5) 2_756 . . . ?
 C1 C2 C3 C4 172.7(3) ?
 C2 C3 C4 C2 0.9(5) . . . 2_756 ?
 C5 C1 C7 C11 -56.0(3) ?
 C6 C1 C7 C11 60.7(3) ?
 C2 C1 C7 C11 -179.3(2) ?
 C5 C1 C7 C8 -175.9(2) ?
 C6 C1 C7 C8 -59.2(3) ?
 C2 C1 C7 C8 60.7(3) ?
 C5 C1 C7 C13 59.8(3) ?
 C6 C1 C7 C13 176.5(2) ?
 C2 C1 C7 C13 -63.5(3) ?
 C11 C7 C8 C9 155.1(3) ?
 C13 C7 C8 C9 37.0(4) ?
 C1 C7 C8 C9 -84.4(3) ?
 C11 C7 C8 C10 -31.6(4) . . . 2_666 ?
 C13 C7 C8 C10 -149.7(3) . . . 2_666 ?
 C1 C7 C8 C10 88.9(3) . . . 2_666 ?
 C10 C8 C9 C10 -1.2(5) 2_666 . . . ?
 C7 C8 C9 C10 172.1(3) ?
 C8 C9 C10 C8 1.2(5) . . . 2_666 ?
 C12 O2 C11 O1 -2.7(4) ?
 C12 O2 C11 C7 176.0(2) ?
 C8 C7 C11 O1 107.5(3) ?
 C13 C7 C11 O1 -129.2(3) ?
 C1 C7 C11 O1 -11.0(4) ?
 C8 C7 C11 O2 -71.2(3) ?
 C13 C7 C11 O2 52.1(3) ?

C1 C7 C11 O2 170.2(2) ?
 C14 O4 C13 O3 -5.5(4) ?
 C14 O4 C13 C7 176.3(2) ?
 C11 C7 C13 O3 -121.8(3) ?
 C8 C7 C13 O3 -1.2(4) ?
 C1 C7 C13 O3 119.2(3) ?
 C11 C7 C13 O4 56.3(3) ?
 C8 C7 C13 O4 176.9(2) ?
 C1 C7 C13 O4 -62.8(3) ?

_refine_diff_density_max 0.221
 _refine_diff_density_min -0.248
 _refine_diff_density_rms 0.056

_shelx_res_file

;

TITL QDM_TCN2_0m_a.res in P-1

QDM_TCN2_0m.res

created by SHELXL-2018/3 at 08:17:14 on 20-Sep-2021

REM Old TITL QDM_TCN2_0m in P-1

REM SHELXT solution in P-1

REM R1 0.189, Rweak 0.003, Alpha 0.044, Orientation as input

REM Formula found by SHELXT: C14 N2 O4

CELL 1.54178 7.0368 7.8604 12.1500 80.096 86.579 70.638

ZERR 2.000 0.0028 0.0030 0.0042 0.013 0.014 0.015

LATT 1

SFAC C H N O

UNIT 28 20 4 8

TEMP -123.150

SIZE 0.18 0.04 0.01

L.S. 20

BOND \$H

ACTA

CONF

LIST 6

FMAP 2

PLAN 20

WGHT 0.070700 0.069300

FVAR 1.31589

O1 4 0.665464 0.567051 0.134029 11.00000 0.05217 0.03699 =
 0.04204 -0.00500 0.00264 -0.01637

O2 4 0.369325 0.517980 0.178985 11.00000 0.04422 0.04029 =
 0.04173 0.00102 -0.00237 -0.00810

O3	4	0.472277	0.109529	0.337739	11.00000	0.05512	0.04651 =
		0.04684	-0.00874	0.00802	-0.02631		
O4	4	0.651070	0.132189	0.179346	11.00000	0.05146	0.03609 =
		0.03896	-0.01340	0.00515	-0.01806		
N1	3	1.078237	0.155369	0.124864	11.00000	0.04442	0.04785 =
		0.04850	-0.01211	0.00514	-0.01072		
N2	3	0.990055	0.563989	0.319098	11.00000	0.05098	0.04038 =
		0.04987	-0.00845	0.00326	-0.01907		
C1	1	0.882888	0.279433	0.301133	11.00000	0.03955	0.02744 =
		0.03581	-0.00727	0.00324	-0.00995		
C2	1	0.955133	0.131678	0.404032	11.00000	0.03249	0.03246 =
		0.04009	-0.00586	-0.00077	-0.00861		
C3	1	0.990591	0.177469	0.504714	11.00000	0.04186	0.03001 =
		0.04170	-0.00567	-0.00387	-0.01183		
AFIX	43						
H3	2	0.985126	0.298929	0.507864	11.00000	-1.20000	
AFIX	0						
C4	1	1.033648	0.047669	0.600041	11.00000	0.04168	0.03300 =
		0.03797	-0.00933	-0.00140	-0.01116		
AFIX	43						
H4	2	1.055720	0.080575	0.668665	11.00000	-1.20000	
AFIX	0						
C5	1	0.985703	0.211977	0.199225	11.00000	0.03866	0.03513 =
		0.03895	-0.00818	0.00563	-0.01184		
C6	1	0.942237	0.441691	0.310362	11.00000	0.04042	0.03222 =
		0.03740	-0.00648	0.00328	-0.01103		
C7	1	0.641269	0.343409	0.294523	11.00000	0.03815	0.02824 =
		0.03427	-0.00788	0.00444	-0.00953		
C8	1	0.556594	0.422384	0.401605	11.00000	0.03892	0.02935 =
		0.03592	-0.00677	0.00325	-0.00934		
C9	1	0.534114	0.314665	0.500922	11.00000	0.04387	0.03070 =
		0.03508	-0.00954	0.00553	-0.01182		
AFIX	43						
H9	2	0.555549	0.188342	0.502810	11.00000	-1.20000	
AFIX	0						
C10	1	0.480129	0.390972	0.598158	11.00000	0.04518	0.03104 =
		0.03436	-0.00535	0.00294	-0.00980		
AFIX	43						
H10	2	0.467851	0.314758	0.666043	11.00000	-1.20000	
AFIX	0						
C11	1	0.564047	0.489601	0.191713	11.00000	0.03553	0.03028 =
		0.03957	-0.01053	0.00207	-0.00722		
C12	1	0.264949	0.661314	0.089871	11.00000	0.05345	0.04469 =
		0.04714	0.00288	-0.01161	-0.00335		

AFIX 137

H12A	2	0.353595	0.661127	0.024512	11.00000	-1.50000
H12B	2	0.227680	0.779960	0.115222	11.00000	-1.50000
H12C	2	0.143116	0.639610	0.069725	11.00000	-1.50000

AFIX 0

C13	1	0.575705	0.179941	0.277463	11.00000	0.03868	0.03179 =
		0.03531	-0.00482	-0.00223	-0.01109		

C14	1	0.619241	-0.026342	0.148418	11.00000	0.05162	0.03779 =
		0.05084	-0.01439	0.00162	-0.01985		

AFIX 137

H14A	2	0.677722	-0.044915	0.074409	11.00000	-1.50000
H14B	2	0.474427	-0.006839	0.146553	11.00000	-1.50000
H14C	2	0.684050	-0.134472	0.203379	11.00000	-1.50000

AFIX 0

HKLF 4

REM QDM_TCN2_0m_a.res in P-1

REM wR2 = 0.1697, GooF = S = 1.022, Restrained GooF = 1.022 for all data

REM R1 = 0.0579 for 1505 Fo > 4sig(Fo) and 0.0997 for all 2404 data

REM 183 parameters refined using 0 restraints

END

WGHT 0.0717 0.0658

REM Highest difference peak 0.221, deepest hole -0.248, 1-sigma level 0.056

Q1	1	0.3519	0.2969	0.6460	11.00000	0.05	0.22
Q2	1	0.3222	0.4741	0.4188	11.00000	0.05	0.21
Q3	1	0.6511	0.2489	0.2900	11.00000	0.05	0.21
Q4	1	0.2344	0.6430	0.2815	11.00000	0.05	0.20
Q5	1	1.0813	0.1027	0.7795	11.00000	0.05	0.20
Q6	1	0.9595	0.2767	0.0564	11.00000	0.05	0.19
Q7	1	0.7275	0.0738	0.6219	11.00000	0.05	0.19
Q8	1	0.7635	0.1855	0.1668	11.00000	0.05	0.18
Q9	1	0.4609	0.1554	0.4474	11.00000	0.05	0.18
Q10	1	0.8779	0.4847	0.3934	11.00000	0.05	0.17
Q11	1	0.2879	0.4346	0.2506	11.00000	0.05	0.17
Q12	1	0.9907	0.0933	0.3982	11.00000	0.05	0.17
Q13	1	0.0790	0.7427	0.1046	11.00000	0.05	0.17
Q14	1	0.9980	0.0261	0.2186	11.00000	0.05	0.17
Q15	1	0.9350	-0.0177	0.1569	11.00000	0.05	0.17
Q16	1	0.8838	0.3706	0.2980	11.00000	0.05	0.17
Q17	1	1.1056	0.0397	0.2214	11.00000	0.05	0.17

Q18	1	0.9093	-0.2142	0.1099	11.00000	0.05	0.16
Q19	1	0.9022	-0.2663	0.2185	11.00000	0.05	0.16
Q20	1	0.6007	0.0760	0.4150	11.00000	0.05	0.16

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_shelx_res_checksum 99852