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Noncatalytic *C*-Amidoalkylation of Acetylacetone and Chromium Acetylacetonate with *N*-Sulfonyl Polychloroacetaldehyde Imines

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Abstract—*N*-(2,2,2-Trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides reacted with acetylacetone and chromium(III) acetylacetonate in the absence of a catalyst to give previously unknown β-aminoketone derivatives, *N*-(3-acetyl-1-polychloro-4-oxopentan-2-yl)arenesulfonamides.

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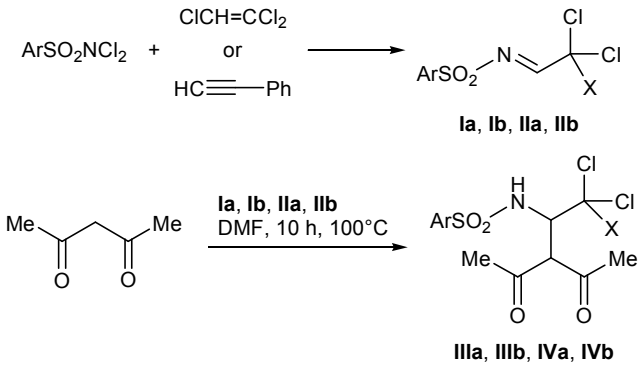
Halogen-substituted aldehyde and ketone imines are important representatives of Schiff bases. The presence in their molecules of two highly electrophilic centers, electron-deficient carbon atom of the azomethine fragment and halomethyl carbon atom, makes such imines very reactive toward nucleophiles, as well as toward aromatic and heteroaromatic compounds, which opens efficient synthetic routes to various functionalized acyclic and heterocyclic derivatives [1–11].

Research works on the reactivity of imines derived from polyhalocarbonyl compounds remain topical. It is known [12–17] that reactions of Schiff bases with 1,3-dicarbonyl compounds acting as carbon-centered nucleophiles provide highly efficient methods for the preparation of practically useful aminocarbonyl compounds which exhibit biological activity and attract interest as subjects for stereochemical and theoretical studies [18]. However, such reactions of 1,3-dicarbonyl compounds with *N*-sulfonyl imines of polyhalocarbonyl compounds have not been reported.

In the present work we examined reactions of *N*-(2,2,2-trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides **I** and **II** with acetylacetone and chromium(III) acetylacetonate with a view to develop synthetic approaches to new *N*-substituted halogen derivatives of aminocarbonyl compounds.

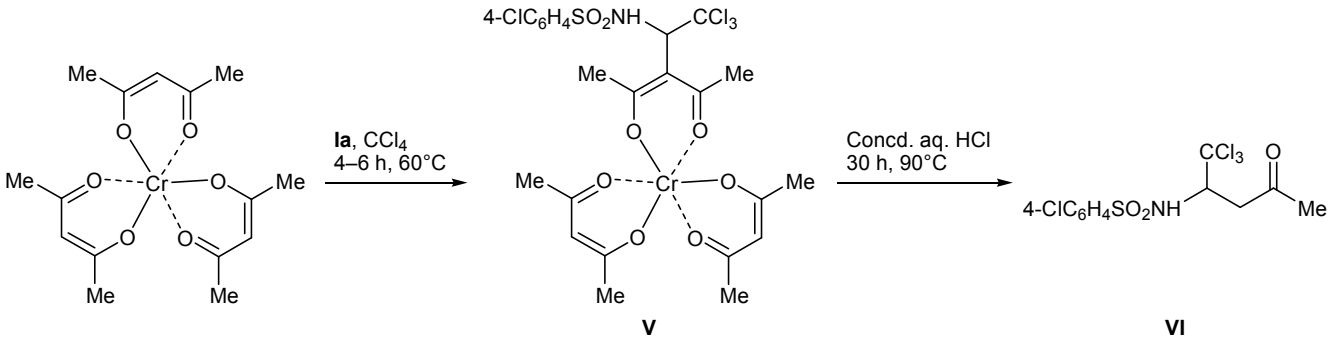
N-Sulfonyl imines **I** and **II** are highly electrophilic activated halogenated aldimines which have become accessible due to development of methods for their preparation by radical reactions of *N*,*N*-dichloroarenesulfonamides with trichloroethylene and phenylacetylene [1, 19–21] (Scheme 1). The best yields of adducts **IIIa**, **IIIb**, **IVa**, and **IVb** were achieved by heating the reactants in dimethylformamide for 10 h at 100°C. The reaction required no any catalytic system, though such systems are commonly used [12–17] to accomplish analogous transformations in the series of Schiff bases derived from aromatic, aliphatic, or functionalized aldehydes. The yields of **IIIa**, **IIIb**, **IVa**, and **IVb** in other solvents or under solvent-free conditions, as well

Scheme 1.



I, **III**, X = Cl; **II**, **IV**, X = Ph; Ar = 4-ClC_6H_4 (**a**), 4-MeC_6H_4 (**b**).

Scheme 2.



as in the reactions carried out at room temperature, were considerably lower.

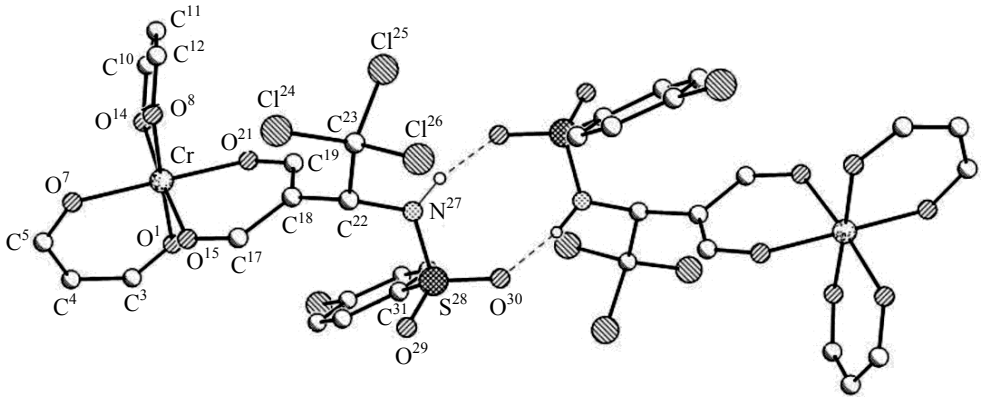
The structure of sulfonamides **IIIa**, **IIIb**, **IVa**, and **IVb** was unambiguously proved by ^{1}H and ^{13}C NMR and IR spectra and elemental analyses. Their ^{1}H NMR spectra contained signals from aromatic protons and those typical of the NHCHCH fragment, namely a doublet of doublets at δ 5.02–5.34 ppm from the NCH proton, a doublet at δ 4.36–4.37 ppm (^3J = 2.4–2.7 Hz) from the CH group located between the carbonyl groups, and a downfield doublet at δ 6.9–8.2 ppm (^3J = 9.1–9.2 Hz) from the NH proton. The methyl and carbonyl groups in the acetylacetone fragment are magnetically nonequivalent, and two sets of the corresponding signals are observed in the ^{1}H and ^{13}C NMR spectra in $CDCl_3$ and DMSO-d_6. The presence in the ^{1}H NMR spectra of a doublet signal from the CH proton with a coupling constant corresponding to spin–spin interaction through three bonds indicates that compounds **IIIa**, **IIIb**, **IVa**, and **IVb** in organic solvents have diketone structure.

Like acetylacetone, metal acetylacetonates can also be involved in reactions with activated halogenated acetaldehyde imines. As an example, Schiff base **Ia** reacted with chromium(III) acetylacetonate to give mixed-ligand complex **V** (Scheme 2). Unlike acetylacetone, the reaction of **Ia** with chromium(III) acetylacetonate occurred under milder conditions (CCl_4, 60°C) and was complete in 4–6 h.

The structure of complex **V** was unambiguously determined by X-ray analysis (see figure and table). Crystals of compound **V** are made up of centrosymmetric dimers formed via intermolecular hydrogen bonds N–H···O=S with an N···O distance of 2.961(3) Å. Insofar as the coordination environment of the chromium atom includes different acetylacetonate ligands, the octahedral coordination entity is slightly distorted, and the Cr–O distances range from 1.932(2) to 1.963(2) Å.

Compound **V** displayed in the IR spectrum absorption bands belonging to stretching vibrations of the NH and SO_2 groups, as well as carbonyl stretching vibration bands whose frequencies differed from those characteristic of the initial acetylacetonate. Signals in the NMR spectra of **V** were appreciably broadened due to the presence of paramagnetic chromium atom.

The structure of **V** was also confirmed by chemical transformations. The hydrolysis with aqueous HCl induced decomposition of the complex and cleavage of the β-dicarbonyl fragment with quantitative formation



Structure of complex **V** (dimer) according to the X-ray diffraction data.

Some interatomic distances and bond angles in the crystalline structure of compound **V** (dimer) according to the X-ray diffraction data

Distance	*d*, Å	Angle	ω, deg	Angle	ω, deg
$Cr–O^{15}$	1.9297(19)	$O^{15}CrO^{14}$	88.59(9)	$C^{17}C^{18}C^{22}$	123.3(2)
$Cr–O^{14}$	1.947(2)	$O^{15}CrO^{21}$	86.16(8)	$C^{19}C^{18}C^{22}$	116.4(2)
$Cr–O^{21}$	1.9526(19)	$O^{14}CrO^{21}$	88.00(9)	$O^{21}C^{19}C^{18}$	124.4(3)
$Cr–O^{7}$	1.9548(19)	$O^{15}CrO^{7}$	178.81(9)	$C^{18}C^{19}C^{20}$	122.7(3)
$Cr–O^{1}$	1.959(2)	$O^{14}CrO^{7}$	90.74(9)	$C^{19}O^{21}Cr$	128.94(18)
$Cr–O^{8}$	1.962(2)	$O^{21}CrO^{7}$	92.84(8)	$N^{27}C^{22}C^{18}$	117.8(2)
$O^{1}–C^{3}$	1.269(4)	$O^{15}CrO^{1}$	90.00(9)	$N^{27}C^{22}C^{23}$	108.5(2)
$C^{3}–C^{4}$	1.390(4)	$O^{14}CrO^{1}$	178.30(9)	$C^{18}C^{22}C^{23}$	116.3(3)
$C^{4}–C^{5}$	1.383(5)	$O^{21}CrO^{1}$	90.95(9)	$C^{22}C^{23}Cl^{24}$	114.5(2)
$C^{5}–O^{7}$	1.280(4)	$O^{7}CrO^{1}$	90.65(9)	$C^{22}C^{23}Cl^{26}$	111.7(3)
$O^{8}–C^{10}$	1.263(4)	$O^{15}CrO^{8}$	89.96(8)	$Cl^{24}C^{23}Cl^{26}$	99.6(2)
$C^{10}–C^{11}$	1.392(5)	$O^{14}CrO^{8}$	90.12(9)	$C^{22}C^{23}Cl^{25}$	109.7(2)
$C^{11}–C^{12}$	1.387(5)	$O^{21}CrO^{8}$	175.73(9)	$Cl^{24}C^{23}Cl^{25}$	112.6(2)
$C^{12}–O^{14}$	1.273(4)	$O^{7}CrO^{8}$	91.02(8)	$Cl^{26}C^{23}Cl^{25}$	108.22(19)
$O^{15}–C^{17}$	1.271(3)	$O^{1}CrO^{8}$	90.84(9)	$C^{22}N^{27}S^{28}$	121.15(19)
$C^{17}–C^{18}$	1.418(4)	$C^{3}O^{1}Cr$	127.5(2)	$O^{30}S^{28}O^{29}$	120.17(13)
$C^{18}–C^{19}$	1.428(4)	$O^{1}C^{3}C^{4}$	124.8(3)	$O^{30}S^{28}N^{27}$	107.14(13)
$C^{18}–C^{22}$	1.523(4)	$C^{5}C^{4}C^{3}$	124.8(3)	$O^{29}S^{28}N^{27}$	106.24(13)
$C^{19}–O^{21}$	1.274(3)	$O^{7}C^{5}C^{4}$	125.1(3)	$O^{30}S^{28}C^{31}$	107.88(15)
$C^{22}–N^{27}$	1.467(3)	$C^{5}O^{7}Cr$	127.1(2)	$O^{29}S^{28}C^{31}$	107.54(14)
$C^{22}–C^{23}$	1.551(5)	$C^{10}O^{8}Cr$	126.0(2)	$N^{27}S^{28}C^{31}$	107.25(14)
$C^{23}–Cl^{24}$	1.732(4)	$O^{8}C^{10}C^{11}$	124.3(3)		
$C^{23}–Cl^{26}$	1.752(4)	$C^{12}C^{11}C^{10}$	125.1(3)		
$C^{23}–Cl^{25}$	1.763(3)	$O^{14}C^{12}C^{11}$	124.0(3)		
$N^{27}–S^{28}$	1.616(3)	$C^{12}O^{14}Cr$	126.2(2)		
$S^{28}–O^{30}$	1.420(2)	$C^{17}O^{15}Cr$	130.34(17)		
$S^{28}–O^{29}$	1.430(2)	$O^{15}C^{17}C^{18}$	123.7(2)		
$S^{28}–C^{31}$	1.761(3)	$C^{17}C^{18}C^{19}$	119.8(3)		

of a previously unknown β-amino ketone, 4-chloro-*N*-[3-oxo-1-(trichloromethyl)butyl]benzenesulfonamide (**VI**) (Scheme 2). The ^{1}H NMR spectrum of **VI** contained signals from protons in the *para*-substituted benzene ring, a singlet from the methyl group, and multiplet signals typical of the $NHCHCH_2$ fragment. These findings in combination with the IR data (absorption bands due to SO_2 and NH groups) and elemental analysis provide reliable proof of the assumed structure.

To conclude, we were the first to reveal that *N*-sulfonyl imines derived from trichloroacetaldehyde and dichloro(phenyl)acetaldehyde are capable of reacting with acetylacetone to produce previously unknown β-amino ketone derivatives. The products attract interest as potentially biologically active substances or their precursors and important reagents whose synthetic value is increased due to the presence of a polychloromethyl fragment. We also found that coordination of acetylacetone to transition metals facilitates its reaction with chlorinated *N*-sulfonyl imines. Taking into account that diketone functionalized in such a way can be isolated as individual compound, this technique may be regarded as a methodical approach to activation of β-dicarbonyl compounds toward aldehyde and ketone imines.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.61 and 100.13 MHz, respectively, using tetramethylsilane as

internal reference. The IR spectra were taken in KBr on a Bruker IFS-25 instrument. Imines **Ia**, **Ib**, **IIa**, and **IIb** were synthesized according to the procedures described in [19–21].

***N*-(3-Acetyl-1,1,1-trichloro-4-oxopentan-2-yl)-4-chlorobenzenesulfonamide (IIIa).** A mixture of 3.21 g (0.01 mol) of imine **Ia**, 1.00 g (0.01 mol) of acetylacetone, and 5 mL of DMF was stirred for 10 h at 100°C. The mixture was cooled to room temperature and diluted with 50 mL of water, and the precipitate was filtered off, dried, and purified by reprecipitation from acetone into water. Yield 3.45 g (82%), mp 115–117°C. IR spectrum, ν, cm^{-1}: 3280 (NH); 1705 (C=O); 1343, 1164 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.28 s (3H, CH_3), 2.30 s (3H, CH_3), 4.50 d [1H, C(O)CHC(O), ^3J = 3.0 Hz], 5.02 d.d (1H, NCH, ^3J = 3.0, 9.1 Hz), 7.17 d (1H, NH, ^3J = 9.1 Hz), 7.43 and 7.79 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 29.47 and 32.95 (CH_3), 63.46 [C(O)**C**HS(O)], 68.71 (NCH), 101.42 (CCl_3); 128.60, 128.72, 129.95, 139.40 (C_6H_4); 199.00 (C=O), 205.10 (C=O). Found, %: C 37.02; H 3.06; Cl 33.48; N 3.25; S 7.50. $C_{13}H_{13}Cl_4NO_4S$. Calculated, %: C 37.08; H 3.11; Cl 33.67; N 3.33; S 7.61.

Compounds **IIIb**, **IVa**, and **IVb** were synthesized in a similar way.

***N*-(3-Acetyl-1,1,1-trichloro-4-oxopentan-2-yl)-4-methylbenzenesulfonamide (IIIb)** was synthesized from 3.01 g (0.01 mol) of imine **Ib**. Yield 3.12 g (78%), mp 97–99°C. IR spectrum, ν, cm^{-1}: 3342 (NH); 1730 (C=O); 1328, 1163 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.23 s (3H, CH_3), 2.24 s (3H, CH_3), 2.38 s (3H, CH_3), 4.47 d [1H, C(O)CHC(O), ^3J = 2.6 Hz], 5.02 d.d (1H, NCH, ^3J = 2.6, 9.1 Hz), 7.05 d (1H, NH, ^3J = 9.1 Hz), 7.24 and 7.70 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 21.56 (CH_3), 29.44 (CH_3), 32.76 (CH_3), 63.66 [C(O)**C**HC(O)], 68.61 (NCH), 101.62 (CCl_3); 127.13, 129.45, 137.98, 143.76 (C_6H_4); 199.33 (C=O), 205.07 (C=O). Found, %: C 41.84; H 3.96; Cl 26.47; N 3.41; S 7.87. $C_{14}H_{16}Cl_3NO_4S$. Calculated, %: C 41.96; H 4.02; Cl 26.54; N 3.50; S 8.00.

***N*-(3-Acetyl-1,1-dichloro-4-oxo-1-phenylpentan-2-yl)-4-chlorobenzenesulfonamide (IVa)** was synthesized from 3.63 g (0.01 mol) of imine **IIa**. Yield 3.70 g (80%), mp 140–142°C. IR spectrum, ν, cm^{-1}: 3218 (NH); 1703 (C=O); 1339, 1168 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 2.19 s (3H, CH_3), 2.27 s (3H, CH_3), 4.36 d [1H, C(O)CHC(O), ^3J = 2.4 Hz], 5.17 d.d (1H, NCH, ^3J = 2.4, 9.2 Hz), 6.87 d (1H, NH, ^3J = 9.2 Hz); 7.33 m, 7.57 m, and 7.67 m (9H, H_{arom}). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 29.33 (CH_3), 32.53 (CH_3), 64.27 [C(O)**C**HC(O)], 67.45 (NCH), 95.36 (CCl_2); 127.45, 128.31, 128.51, 129.00, 138.65, 138.83, 139.39, 144.12 (C_{arom}); 200.03 (C=O), 205.12 (C=O). Found, %: C 49.22; H 3.97; Cl 22.85; N 3.15; S 7.01. $C_{19}H_{18}Cl_3NO_4S$. Calculated, %: C 49.31; H 3.92; Cl 22.98; N 3.03; S 6.93.

***N*-(3-Acetyl-1,1-dichloro-4-oxo-1-phenylpentan-2-yl)-4-methylbenzenesulfonamide (IVb)** was synthesized from 3.42 g (0.01 mol) of imine **IIb**. Yield 3.71 g (84%), mp 103–105°C. IR spectrum, ν, cm^{-1}: 3232 (NH); 1702 (C=O); 1333, 1156 (SO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.81 s (3H, CH_3), 2.16 (3H, CH_3), 2.33 s (3H, CH_3), 4.37 d [1H, C(O)CHC(O), ^3J = 7.1 Hz], 5.34 d.d (1H, NCH, ^3J = 7.1, 10.0 Hz); 7.20 m, 7.28 m, 7.48 m, and 7.56 m (9H, H_{arom}); 8.22 d (1H, NH, ^3J = 10.0 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 20.74 (CH_3), 29.06 (CH_3), 31.02 (CH_3), 64.05 [C(O)**C**HC(O)], 67.34 (NCH), 96.13 (CCl_2); 125.86, 127.32, 128.02, 128.71, 129.49, 138.23, 139.12, 141.78 (C_{arom}); 199.19 (C=O), 200.72 (C=O). Found, %: C 54.22; H 4.67; Cl 15.95; N 3.05; S 7.18. $C_{20}H_{21}Cl_2NO_4S$. Calculated, %: C 54.30; H 4.79; Cl 16.03; N 3.17; S 7.25.

{3-[1-(4-Chlorobenzenesulfonamido)-2,2,2-trichloroethyl]pentane-2,4-dionato}bis(acetylacetonato)chromium(III) (V). A mixture of 3.21 g (0.01 mol) of imine **Ia** and 3.49 g (0.01 mol) of chromium(III) acetylacetonate in 25 mL of carbon tetrachloride was stirred for 6 h at 60°C in a stream of argon. The precipitate was filtered off and recrystallized from carbon tetrachloride–acetone (4:1). Yield 5.18 g (77%), mp 143–145°C. IR spectrum, ν, cm^{-1}: 3227 (NH); 1588 (C=O); 1526 (C=C); 1348, 1149 (SO_2). Found, %: C 41.36; H 4.23; Cl 21.22; N 2.21; S 4.54. $C_{23}H_{32}Cl_4CrNO_8S$. Calculated, %: C 41.03; H 4.34; Cl 21.06; N 2.08; S 4.76.

4-Chloro-*N*-(1,1,1-trichloro-4-oxopentan-2-yl)-benzenesulfonamide (VI). A mixture of 6.73 g (0.01 mol) of compound **V** and 25 mL of concentrated aqueous HCl was stirred for 30 h at 90°C. The precipitate was filtered off, washed with water, and dried under reduced pressure. Yield 3.71 g (98%), mp 204–206°C. IR spectrum, ν, cm^{-1}: 3243 (NH); 1583 (C=O); 1350, 1164 (SO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 1.87 s (3H, CH_3), 2.72 and 3.12 (2H, CH_2, *ABX*, ^3J = 6.6, 3.0, ^2J = 18.1 Hz), 4.57 m (1H, CH, ^3J = 6.6, 3.0, 8.9 Hz), 7.64 and 7.76 (4H, *AA′BB′*, C_6H_4), 8.85 d (1H, NH, ^3J = 8.9 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 29.50 (CH_3), 45.14 (CH_2), 63.49 (NCH), 102.68 (CCl_3); 128.22, 129.02, 137.26, 140.30

(C_6H_4); 202.94 (C=O). Found, %: C 34.78; H 2.99; Cl 37.63; N 3.84; S 8.75. $C_{11}H_{11}Cl_4NO_3S$. Calculated, %: C 34.85; H 2.92; Cl 37.41; N 3.69; S 8.46.

X-Ray diffraction study of compound V. Reflection intensities from a single crystal of complex **V** were measured on a Bruker Smart Apex CCD diffractometer at 240 K (MoK_α radiation, λ 0.71073 Å). Absorption by the crystal was taken into account using SADABS program. The structure was solved by the direct method and was refined by the full-matrix least-squares method in anisotropic approximation for all non-hydrogen atoms. The positions of hydrogen atoms were calculated geometrically and were refined according to the riding model. Difference syntheses of electron density revealed peaks corresponding to a solvent molecule, but we failed to localize it reliably; therefore, it was excluded from the refinement in the final step (SQUEEZE function in PLATON program [22]). All calculations were performed using Bruker SHELXTL Version 6.14. Crystallographic parameters of compound **V**: triclinic crystal system, space group *P*-1; a = 7.6101(4), b = 11.6074(7), c = 18.7245(11) Å; α = 101.136(3), β = 101.675(3), γ = 97.682(3)°; V = 1563.4(2) Å^3; Z = 2; d_{calc} = 1.424 g/cm^3; μ = 0.816 mm^{-1}; 1.14 < θ < 28.29°. Total of 25921 reflection intensities were measured, 7650 of which were independent (R_{int} = 0.0674) and 7909 reflections were characterized by $I > 2\sigma(I)$; 361 refined parameters; goodness of fit 0.827; final divergence factors R_1 = 0.0497, wR_2 = 0.1135 for reflections with $I > 2\sigma(I)$, R_1 = 0.1155, wR_2 = 0.1266 for all reflections (I_{hkl}); maximum and minimum residual electron density 0.818/–0.483 e Å^{-3}.

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