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Efficient one-pot preparation of *bis*alkyl xanthogen disulfides from alcohols

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An efficient one-pot synthetic procedure for the preparation of *bis*alkyl xanthogen disulfides is achieved via oxidation of the *in situ* generated xanthate salt in an aqueous condition using half an equivalent of molecular iodine in one pot.

Keywords: disulfide; xanthate; xanthogen; iodine; one pot

1. Introduction

*Bis*alkyl xanthogen disulfides is an important class of compounds that has found diverse applications as an intermediate for free radical polymerization that allows control of molecular weight and polydispersity of polymers, a process well known as Reversible Addition Fragmentation Chain Transfer or RAFT mediated polymerization (*1*). These compounds are valuable synthetic intermediates and have shown numerous applications as pesticides and fungicides in agriculture and sulfur vulcanization in rubber manufacturing (*2*). Introduction of xanthogen disulfides in the polymerization of polychloroprene latex adhesive gives the latter excellent heat resistance and green bond strength (*3*) as well as good colloidal stability (*4*). 1,3-Dithiol-2-one, precursors of tetrathiafulvalene that are extensively used in conjugation with tetracyano-*p*-quinodimethane (TCNQ) as organic conductors possessing unusual high electrical conductivity, are prepared in a single step from diisopropyl xanthogen disulfide and alkyne (*5*). Diisopropyl xanthogen disulfide has also found extensive applications in the rubber tanning industry as a chain-transfer agent in free radical emulsion polymerization of butadiene styrene rubber, as well as in the preparation of Neoprene rubber, resistant to scorch and aging (*6*). Together with bis-(amidophenyl) polysulfides, it is also used in the preparation of xanthogen group terminated oligomers like Telichelic oligobutadienes (*7*).

However, the chemistry of dialkyl xanthogen disulfides remains largely unexplored and only a few examples of its preparation have been reported in the literature. Xanthogen disulfides are

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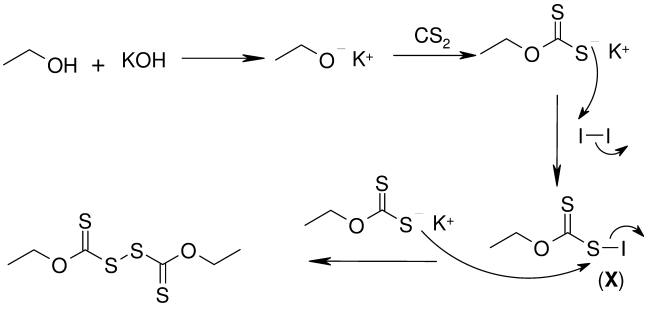
prepared mostly by oxidation of xanthate salts. The commonly used oxidizing agent includes chloramine-T (*8*), Ph_2TeCl_2 or telluracyclopentane-1,1-diiodide (*9*), Cl_2 gas under basic medium (*10*), bromonitromethane (*11*), as well as concentrated iodine in the presence of KOH (*12*). Xanthogen disulfides have recently been prepared from xanthate salt using *p*-tosyl chloride under basic condition (*13*). The inherent difficulty in the preparation of dialkyl xanthogen disulfides is due to the sensitivity of the oxidation reaction to impurities and the need for very specific reaction conditions. Iodine, a cheap oxidant (*14*), has been employed for this purpose (*15*) but to a very limited number of substrates because of the difficulties in the isolation of the intermediate xanthate salt. Again, this method is a two-step process involving the isolation of the prepared xanthate salts followed by oxidation with concentrate iodine under strongly basic conditions.

In this article, we disclose a robust procedure to synthesize xanthogen disulfides in one pot from the *in situ* generated xanthate salts and molecular iodine. The xanthate salts were prepared by the reaction of alcohol (**1**) and CS_2 in the presence of KOH pellets following the modified literature procedure (*16*). The best result is obtained when a small quantity of acetonitrile was used as the solvent. The solvent dimethylsulfoxide (DMSO) also worked equally well but acetonitrile was preferred due to ease of workup. To this xanthate salt molecular iodine was added to obtain the xanthogen disulfides (**1a)** in one pot. The expected xanthogen disulfide (**1a)** was confirmed by HRMS analysis 243.3873 (Calcd. mass for $C_6H_{10}O_2S_4 = 243.4157$) and elemental analysis. IR spectrum revealed a characteristic peak at 1021 cm^{-1} (C=S), ^1H NMR spectrum showed signals at δ 1.42 (t, 6H, $J = 7.2$ Hz, CH_3), 4.70 (q, 4H, $J = 7.2$ Hz, O−CH_2) and ^{13}C NMR at δ 13.8, 71.8, 207.9 (C=S). The proposed mechanism of formation of xanthogen disulfide is shown in Scheme 1. The xanthate salt attacks iodine, giving the intermediate (X), which is in turn attacked by a second molecule of xanthate displacing iodide. The requirement of one half the equivalent of iodine is evident from the reaction mechanism.

The success of this methodology was applied for the preparation of diisopropyl xanthogen disulfide (**2a**) and bis(2-methyl propyl) xanthogen disulfide (**3a**) in good yields of 78% and 75%, respectively (Table 1), following the similar synthetic procedure. The formation of the desired products (**2a**) and (**3a**) were confirmed by their HRMS analysis 271.9706 (Calcd. mass for $C_8H_{14}O_2S_4 = 271.4605$) and 299.7759 (Calcd. mass for $C_{10}H_{18}O_2S_4 = 299.5141$), respectively. Elemental analysis of (**2a**) and (**3a**) further supports the desired formulation. IR spectrum revealed a characteristic band at 1005 and 1026 cm^{-1}, respectively, which corresponds to (C=S). ^1H NMR and ^{13}C NMR spectrum showed characteristic signals supporting the proposed structure (*c.f.* Table 1).

This one-pot methodology was further extended to the preparation of didecyl xanthogen disulfide (**4a**) in good yields from decanol (**4**), a substrate containing a long alkyl chain. The structure of the product (**4a**) has been supported by spectral and analytical data (Table 1). The proposed methodology was further applied to yet another secondary alcohol, 4-*tert*-butylcyclohexanol (**5**),



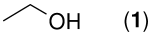
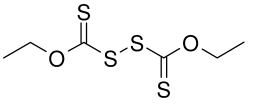
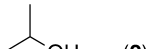
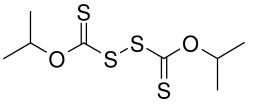
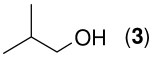
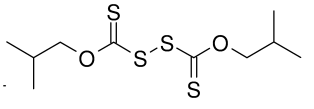
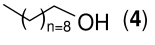
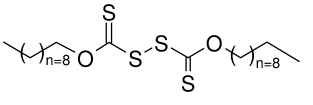
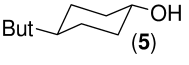
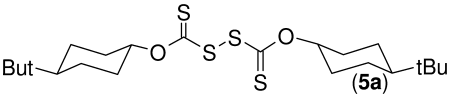
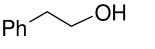
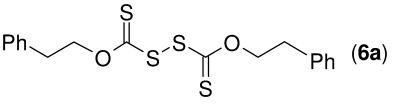
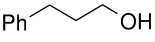
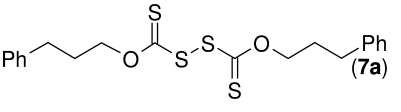
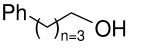
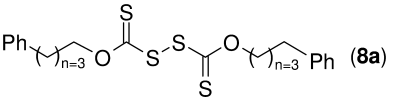
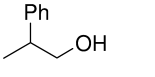
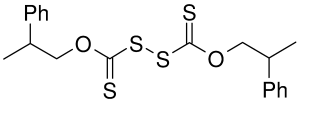
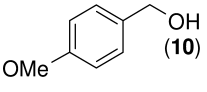
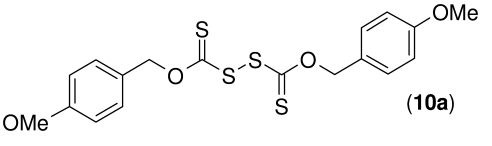
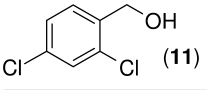
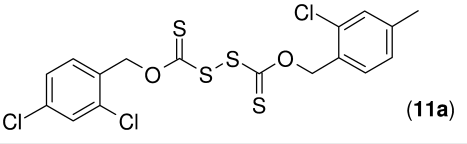
Scheme 1. Proposed mechanism for the formation of xanthogen disulfide.

for the synthesis of bis(4-*tert*-butyl cyclohexanol) xanthogen disulfide (**5a**). The product (**5a**) was found to be a needle-like white crystalline solid, m.p. 124°C. The formation of the desired product was confirmed by spectral and analytical data (Table 1). This synthetic protocol was further extended to aromatic primary alcohols (**6**–**9**) (Table 2) for the preparation of their respective xanthogen disulfide in good yields. All the predicted products gave expected spectral and analytical data (Table 1).

Table 1. Spectral and analytical data.

Entry	Spectral data
1a (*13*)	^{1}H NMR ($CDCl_3$): δ 1.42 (t, 6H, $J = 7.2$ Hz, CH_3), 4.70 (q, 4H, $J = 7.2$ Hz, O−CH_2). ^{13}C NMR ($CDCl_3$)δ 13.8, 71.8, 207.9 (C=S); IR (KBr): 2981 (m), 1460 (m), 1440 (m), 1368 (m), 1242 (s), 1108 (m), 1021 (s), 847 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 243.3873, $C_6H_{10}O_2S_4$ requires 243. 4157. Calcd. C 59.21, H 4.14, S 52.69%; found: C 59.32, H 4.09, S 52.78%.
2a (*9,22*)	^{1}H NMR ($CDCl_3$): δ 1.40 (d, 12H, $J = 3.2$ Hz, $2 \times CH_3$), 5.62–5.65 (m, 2H, O−CH). ^{13}C NMR ($CDCl_3$)δ 21.3, 80.4, 207.1 (C=S); IR (KBr): 2981 (m), 2934 (w), 1372 (m), 1352 (m), 1266 (s), 1087 (s), 1006 (s), 898 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 271.9706 (Calcd. mass for $C_8H_{15}O_2S_4 = 271.4605$). Calcd. C 35.39, H 5.57, S 47.25%; found: C 35.26, H 5.62, S 47.38%.
3a (*23*)	^{1}H NMR ($CDCl_3$): δ 1.0 (d, 12H, $J = 6.8$ Hz, $2 \times CH_3$), 2.10–2.17 (m, 2H, CH), 4.36 (d, 4H, $J = 7.2$ Hz, O–CH_2). ^{13}C NMR ($CDCl_3$)δ 19.1, 27.8, 81.3, 207.1 (C=S); IR (KBr) 2963 (m), 1468 (m), 1371 (m), 1263 (s), 1026 (s), 964 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 299.7759 (Calcd. mass for $C_{10}H_{19}O_2S_4 = 299.5141$). calcd. C 40.09, H 6.39, S 42.82%; found: C 40.19, H 6.35, S 42.78%.
4a	^{1}H NMR ($CDCl_3$): δ 0.89 (t, 6H, $J = 6$ Hz, CH_3), 1.24-1.40 (m, 28H, $7 \times CH_2$), 1.76–1.80 (m, 4H, CH_2), 4.59 (t, 4H, $J = 6$ Hz, O–CH_2). ^{13}C NMR ($CDCl_3$): δ 14.3, 22.9, 26.0, 28.3, 29.4, 29.5, 29.7, 32.1, 75.9, 207.5 (C=S); IR (KBr): 2925 (s), 2855 (s), 1639 (w), 1464 (m), 1384 (m), 1260 (s), 1023 (s), 909 (w), 722 (w) cm^{-1}. HRMS (ESI): MH$^+$ found 467.6388 (Calcd. mass for $C_{22}H_{43}O_2S_4 = 467.8355$). Calcd. C 56.48, H 9.26, S 27.42%; found: C 56.55, H 9.18, S 27.09%.
5a	M.p. 124^{o}C. ^{1}H NMR ($CDCl_3$): δ 0.84 (s, 18 H, $3 \times CH_3$), 0.99–1.18 (m, 6H), 1.41–1.50 (dd, 4H, CH_2), 1.84 (d, 4H, $J = 10.8$ Hz, CH_2), 2.14 (d, 4H, $J = 9.6$ Hz, CH_2), 5.28–5.36 (m, 2H, CH). ^{13}C NMR ($CDCl_3$): δ 25.6, 27.8, 31.4, 32.5, 47.0, 86.0, 207.1; IR (KBr): 2946 (m), 1468 (m), 1365 (m), 1326 (m), 1261 (s), 1014 (s), 901 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 463.7841 (Calcd. mass for $C_{22}H_{39}O_2S_4 = 463.7999$). Calcd. C 56.97, H 8.47, S 27.65%; found: C 56.99, H 8.51, S 27.44%.
6a	^{1}H NMR ($CDCl_3$): δ 3.00 (t, 4H, $J = 6.8$ Hz, CH_2), 4.67 (t, 4H, $J = 6.8$ Hz, O–CH_2), 7.12–7.29 (m, $10 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$)δ 34.4, 75.6, 126.7, 128.6, 128.9, 136.8, 207.0 (C=S); IR (KBr): 3063 (m), 3028 (m), 2950 (m), 1496 (m), 1455 (m), 1384 (m), 1259 (s), 1025 (s), 949 (m), 748 (m), 698 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 395.6624 (Calcd. mass for $C_{18}H_{19}O_2S_4 = 395.5983$). Calcd. C 54.65, H 4.84, S 32.42%; found: C 54.58, H 4.80, S 31.98%.
7a	^{1}H NMR ($CDCl_3$): δ 2.07–2.13 (m, 4H, CH_2), 2.7 (t, $J = 7.2$ Hz, 4H, CH_2), 4.59 (t, 4H, $J = 6.4$ Hz, O–CH_2), 7.14-7.21 (m, 6H, $3 \times$ Ar–CH), 7.25–7.29 (m, 4H, $2 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$): δ 29.9, 32.1, 74.7, 126.4, 128.6, 128.7, 140.5, 207.3; IR (KBr): 3026 (m), 2951 (m), 1496 (m), 1455 (m), 1259 (s), 1021 (s), 746 (m), 699 (s) cm^{-1}. HRMS (ESI): M+2H$^+$ found 424.1492 (Calcd. mass for $C_{20}H_{24}O_2S_4$= 424.5693). Calcd. C 48.57, H 4.89, S 25.93%; found: C 48.60, H 4.94, S 25.72%.
8a	^{1}H NMR ($CDCl_3$): δ 1.62–1.68 (m, 4H, CH_2), 1.72–1.76 (m,4H, CH_2), 2.59 (t, $J = 7.2$ Hz, 4H, CH_2), 4.50 (t, 4H, $J = 6.0$ Hz, O−CH_2), 7.12–7.19 (m, $6 \times$ Ar–CH), 7.24–7.28 (m, $4 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$): δ 27.91, 27.94, 35.58, 75.73, 126.25, 128.71, 141.87, 207.47 (C=S); IR (KBr): 3025 (m), 2943 (m), 1454 (m), 1385 (m), 1265 (s), 1018 (s), 747 (m), 699 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 479.7468 (Calcd. mass for $C_{24}H_{31}O_2S_4 = 479.7577$) Calcd. C 60.08, H 6.51, S 26.73%; found: C 60.19, H 6.42, S 26.65%.
9a	^{1}H NMR ($CDCl_3$): δ 1.31 (dd, 6H, CH_3), 3.17–3.23 (m, 2H, CH), 4.4– 4.6 (m, 4H, O−CH_2), 7.25 (m, 10H, $5 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$): δ 17.8, 38.8, 80.2, 127.2, 127.6, 129.0, 142.3, 207.1 (C=S); IR (KBr) 1028 cm^{-1}. HRMS (ESI): MH$^+$ found 423.8815 (Calcd. mass for $C_{20}H_{23}O_2S_4 = 423.6435$). Calcd. C 56.70, H 5.47, S 30.27%; found: C 56.64, H 5.56, S 29.82%.
10a	^{1}H NMR ($CDCl_3$): δ 3.55 (s, 4H, O−CH_2), 3.79 (s, 6H, O−CH_3), 6.84 (d, 4H, $J = 10.4$ Hz, $2 \times$ Ar–CH), 7.19 (d, 4H, $J = 10.4$ Hz, $2 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$): δ 35.2, 55.5, 114.0, 114.2, 130.8, 158.8, 207.2 (C=S); IR (KBr): 2954 (w), 2834 (w), 1613 (s), 1514 (m), 1250 (m), 1175 (m), 1034 (m), 831 (m) cm^{-1}. HRMS (ESI): MH$^+$ found 427.8503 (Calcd. mass for $C_{18}H_{19}O_4S_4 = 427.6010$). Calcd. C 50.56, H 4.48, S 30.00%; found: C 50.60, H 4.52, S 29.85%.
11a	M.p. 89.5 °C; ^{1}H NMR ($CDCl_3$): δ 5.6 (s, 4H, O–CH_2), 7.15–7.38 (m, 6H, $3 \times$ Ar–CH). ^{13}C NMR ($CDCl_3$): δ 72.8, 127.5, 129.7, 130.3, 131.0, 134.6, 135.7, 206.5 (C=S). IR (KBr): 2948 (m), 2862 (m), 1365 (m), 1326 (m), 1261 (s), 1014 (s), 900 (w) cm^{-1}. HRMS (ESI): M$^+$ found 504.9116 (Calcd. mass for $C_{16}H_{10}Cl_4O_2S_4 = 504.321$). Calcd. C 38.10, H 1.99, S 25.43%; found: C 38.14, H 2.02, S 25.24%.

Table 2. Preparation of *bis*alkyl xanthogen disulfides from corresponding alcohols.[a]

Alcohol	Product[b]	Yield (%)[c]
 (**1**)	 (**1a**)	82
 (**2**)	 (**2a**)	78
 (**3**)	 (**3a**)	75
 (**4**)	 (**4a**)	76
 (**5**)	 (**5a**)	68
 (**6**)	 (**6a**)	78
 (**7**)	 (**7a**)	70
 (**8**)	 (**8a**)	75
 (**9**)	 (**9a**)	69
 (**10**)	 (**10a**)	63
 (**11**)	 (**11a**)	72

Notes: [a]Reactions were monitored by TLC.
[b]Confirmed by ^{1}H and ^{13}C NMR.
[c]Isolated yield.

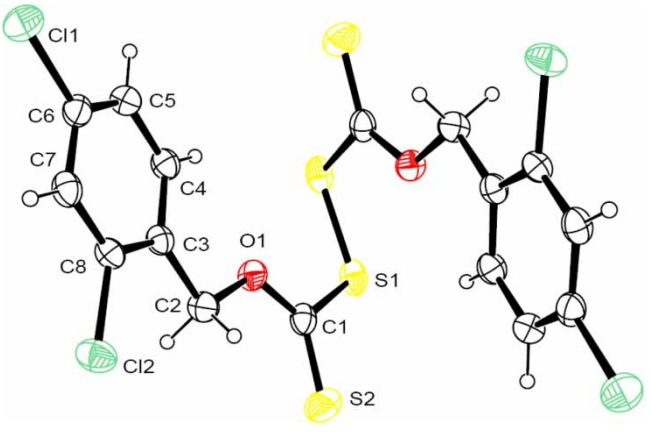


Figure 1. ORTEP view with atom numbering scheme of 11a (*17*).

Benzylic alcohols *p*-methoxy benzylalcohol (**10**) and 2,4-dichloro benzylalcohol (**11**) yielded their corresponding xanthogen disulfides (**10a**) and (**11a**) in satisfactory yields. Structures have been confirmed by spectral and analytical data. Structure of the product (**11a)** has been further confirmed by crystal X-ray crystallography. The ORTEP view with atom numbering scheme of (**11a)** is shown in Figure 1 (*17*).

In this molecule (**11a**) the measured S−S bond distance is 2.054(1) Å, and C1−S2 (C=S) is 1.613(2) Å. The torsion angle between C−S−S−C is found to be 77.90°. The observed S−S bond distance, 2.054(1) Å, is comparable to S−S bond distance of 2.020(1) Å for similar compound dibenzoyldisulfide (*18*). The bond length of disulfides depends upon the value of the torsion angle, which may due to the variation in the loan pair repulsions between two sulfur atoms. Similarly, the measured C1−S1 bond length is 1.767(2) Å, which is slightly longer than C1−S2 1.613(2) Å) because of variations in loan pair repulsions. The bond angle between C−S−S and C1−O1−C2 respectively are 105.72°(7) and is 117.19°(2).

In conclusion, we have demonstrated an efficient method for the preparation of *bis*alky xanthogen disulfides directly from alcohol in one pot. This method is highly efficient and environmentally benign, giving a good yield of products in a shorter reaction time.

2. Experimental

All the reagents were of commercial grade and purified according to established procedures. Organic extracts were dried over anhydrous sodium sulfate. Solvents were removed in a rotary evaporator under reduced pressure. Reactions were monitored by TLC on silica gel 60 F_{254} (0.25 mm). Column chromatography was done with silica gel (60–120 mesh). NMR spectra were recorded in $CDCl_3$ with tetramethylsilane as the internal standard for ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz); the chemical shifts are expressed as δ values (ppm). HRMS spectra were recorded using WATERS MS system, Q–Tof premier and data analyzed using Mass Lynx 4.1. Melting points were recorded in a Buchi B-545 melting point apparatus and are uncorrected. IR spectra were recorded in KBr or neat on a Nicolet Impact 410 spectrophotometer.

2.1. *General procedure for the synthesis of xanthogen disulfides*

An amount of KOH pellets (25 mmol) and alcohol (25 mmol) in CH_3CN (10 mL) were refluxed for 20 min and allowed to cool to room temperature. Carbon disulfide (30 mmol) was added slowly

to the reaction mixture with constant stirring in an ice-cold condition. The resultant xanthate salt was dissolved in water (5–10 mL) to which iodine (12.5 mmol) was added pinch-wise over a period of 15 min. Solvent CH_3CN was removed in a rotary evaporator and the product was extracted from hexane (2×20 mL). The hexane layer was washed with 5% sodium thiosulphate solution (2×5 mL). The hexane layer was dried over anhydrous sodium sulphate and removed under reduced pressure. Further purification was achieved by passing it through a short column of silica gel (100% hexane) to obtain the products in good yield.

3. Crystallographic description

Crystal data were collected with a Bruker Smart Apex-II CCD diffractometer using graphite monochromated MoKα radiation ($\lambda = 0.71073$ Å) at 298 K. Cell parameters were retrieved using SMART (*19*) software and refined with SAINT (*19*) on all observed reflections. Data reduction was performed with the SAINT software and corrected for Lorentz and polarization effects. Absorption corrections were applied with the program SADABS (*20*). The structure was solved by direct methods implemented in SHELX-97 (*21*) program and refined by full-matrix least-squares methods on F2. All non-hydrogen atomic positions were located in difference Fourier maps and refined anisotropically. The hydrogen atoms were placed in their geometrically generated positions. The yellowish crystal was isolated in rectangular shapes from ethylacetate : acetonitrile (9 : 1) at room temperature. CCDC numbers for compound **11a** is CCDC 695818. This data can be obtained from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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(17) Crystallographic description of bis(2,4-dichloro benzyl) xanthogen disulfide (**11a**) Crystal dimension (mm), 0.31 × 0.24 × 0.18, $C_{16}H_{10}O_2Cl_4S_4$, $M_r = 504.28$; Monoclinic, Space group C2/c; a = 16.3982(4) Å, b = 6.0027(2) Å, c = 21.3604(7) Å, $\alpha = ß = 90°$, $\gamma = 102.329(3)°$, V = 2054.09(11) Å, $^3Z = 4$, $\rho_{cal} = 1.631\,Mg/m^3$; reflection collected/unique = 6143/1730; refinement method = $full$ matrix least squares on f^2; final R indices[$I > 2\sigma_1$] $R_1 = 0.0285$, $wR_2 = 0.0683$, R indices(all data) $R_1 = 0.0344$, $wR_2 = 0.0728$; goodness of fit = 1.045.
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