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Synthesis of New Heterocyclic System of 4,5,7,8-Tetrahydroimidazo[1,2-*c*][1,3]thiazolo [4,5-*e*][1,3,2]diazaphosphinine Starting from 2-Aroylamino-3,3-dichloroacrylonitrile

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ABSTRACT: *Easily accessible 2-aroylamino-3,3-dichloroacrylonitriles, when treated successively with ethylenediamine, phosphorus pentasulfide, water, and methyl and benzyl halides, furnish the corresponding derivatives of 4,5,7,8-tetrahydroimidazo[1,2-c][1,3]thiazolo[4,5-e][1,3,2]diazaphosphinine, a novel fused heterocycle. The structure of the compounds obtained is unequivocally confirmed by the spectroscopic method and X-ray diffraction analysis.* © 2010 Wiley Periodicals, Inc. Heteroatom Chem 21:492–498, 2010; View this article online at wileyonlinelibrary.com. DOI 10.1002/hc.20638

INTRODUCTION

2-Aroylamino-3,3-dichloroacrylonitriles that are readily prepared from easily accessible chloral adducts of carboxamides [1–4] have been extensively utilized in heterocyclic synthesis [3–18]. The present study offers their new application as starting materials to obtain a hitherto unknown heterocyclic system, 4,5,7,8-tetrahydroimidazo[1,2-*c*][1,3]thiazolo[4,5-*e*][1,3,2]diazaphosphinine.

RESULTS AND DISCUSSION

2-Aroylamino-3,3-dichloroacrylonitriles react with N-nucleophiles (primary aliphatic and aromatic amines) to yield, as a rule, 5-amino-4-cyanooxazole derivatives [3–10]. Interestingly, the reaction with 1,4-*N*,*N*-dinucleophiles, for example, ethylenediamine, proceeds in quite a different way: Instead of cyclization to 5-aminooxazole derivatives, two chlorine atoms in reagents **1a–c** are substituted by the ethylenediamine residue thus affording the imidazolidine ring as in products **2a–c** (Scheme 1 and Table 1).

The IR spectra of compounds **2a–c** show the valence vibration bands of the carbonyl and cyano groups in the respective regions 1652–1657 and 2155–2177 cm^{-1} (Table 2). ^1H NMR absorption is represented by two singlets of the imidazolidine NH groups at 6.56–6.85 ppm and the signals of the amide NH group at 8.66–8.92 ppm as well as the resonances of aliphatic and aromatic protons, with

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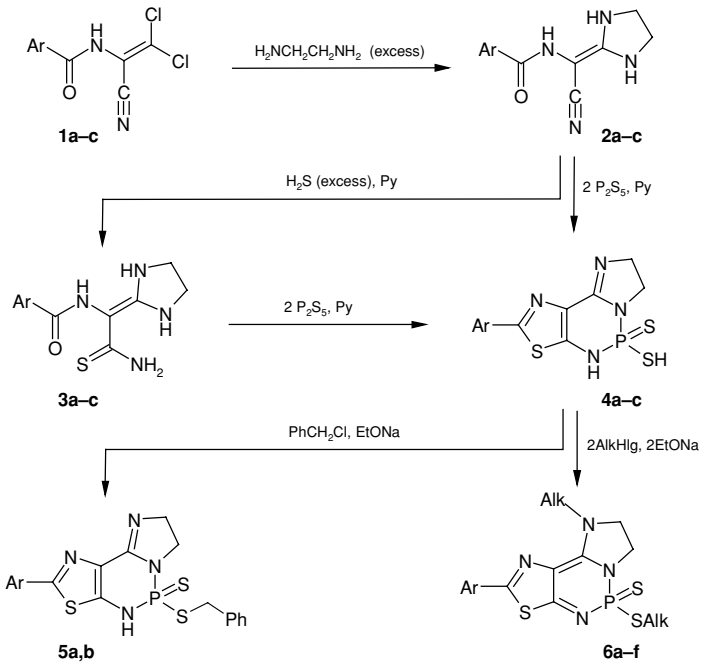
TABLE 1 Physical and Analytical Data of Compounds **2–7**

Compound (Product)	*M.p. (°C)*	*Yield (%)*	*Molecular Formula (Weight)*	*Elemental Analysis, Found (Calcd) (%)*				
				C	*H*	*N*	*P*	*S*
2a	213–215	52	$C_{12}H_{12}N_4O$(228.26)	63.08 (63.15)	5.22 (5.30)	24.47 (24.45)	–	–
2b	245–247	60	$C_{13}H_{14}N_4O$ (242.28)	64.53 (64.45)	5.75 (5.82)	23.03 (23.12)	–	–
2c	224–226	62	$C_{12}H_{11}ClN_4O$ (262.70)	54.94 (54.87)	4.29 (4.22)	21.38 (21.33)	–	–
3a	245–246	89	$C_{12}H_{14}N_4OS$ (262.34)	59.88 (59.94)	5.32 (5.38)	21.42 (21.36)	–	12.27 (12.22)
3b	261–262	92	$C_{13}H_{16}N_4OS$ (276.36)	56.54 (56.50)	5.90 (5.84)	20.31 (20.27)	–	11.65 (11.60)
3c	253–254	94	$C_{12}H_{13}ClN_4OS$ (296.78)	48.52 (48.57)	4.36 (4.42)	18.93 (18.88)	–	10.84 (10.80)
4a	304–306	71	$C_{12}H_{11}N_4PS_3$ (338.41)	42.63 (42.59)	3.24 (3.28)	16.51 (16.56)	9.21 (9.15)	28.48 (28.42)
4b	338–340	72	$C_{13}H_{13}N_4PS_3$ (352.44)	44.36 (44.30)	3.78 (3.72)	15.85 (15.90)	8.73 (8.79)	27.23 (27.29)
4c	355 (dec.)	75	$C_{12}H_{10}ClN_4PS_3$ (372.86)	38.61 (38.66)	2.75 (2.70)	15.08 (15.03)	8.36 (8.31)	25.84 (25.80)
5a	164–165	61	$C_{19}H_{17}N_4PS_3$ (428.54)	53.19 (53.25)	4.04 (4.00)	13.02 (13.07)	7.28 (7.23)	22.51 (22.45)
5b	240–241	63	$C_{20}H_{19}N_4PS_3$ (442.57)	54.24 (54.28)	4.37 (4.33)	12.63 (12.66)	7.03 (7.00)	21.76 (21.74)
6a	253–254	70	$C_{14}H_{15}N_4PS_3$ (366.47)	45.93 (45.89)	4.09 (4.13)	15.25 (15.29)	8.49 (8.45)	26.30 (26.25)
6b	243–244	71	$C_{15}H_{17}N_4PS_3$ (380.50)	47.39 (47.35)	4.54 (4.50)	14.68 (14.72)	8.10 (8.14)	25.33 (25.28)
6c	223–225	74	$C_{14}H_{14}ClN_4PS_3$ (400.91)	41.90 (41.94)	3.48 (3.52)	13.92 (13.97)	7.68 (7.73)	23.94 (23.99)
6d	186–188	83	$C_{26}H_{23}N_4PS_3$ (518.67)	60.25 (60.21)	4.52 (4.47)	10.84 (10.80)	5.93 (5.97)	18.59 (18.55)
6e	187–189	85	$C_{27}H_{25}N_4PS_3$ (532.69)	60.93 (60.88)	4.78 (4.73)	10.56 (10.52)	5.85 (5.81)	18.10 (18.06)
6f	192–193	86	$C_{26}H_{22}ClN_4PS_3$ (553.11)	56.42 (56.46)	4.06 (4.01)	10.19 (10.13)	5.56 (5.60)	17.38 (17.39)
7a	216–218	85	$C_{20}H_{19}N_4PS_3$ (442.57)	54.22 (54.28)	4.38 (4.33)	12.70 (12.66)	7.03 (7.00)	21.70 (21.74)
7b	241–243	83	$C_{21}H_{21}N_4PS_3$ (456.59)	55.28 (55.24)	4.61 (4.64)	12.32 (12.27)	6.83 (6.78)	21.03 (21.07)

TABLE 2 Spectroscopic Data of Compounds **2–7**

Compound	*IR (KBr) (cm^{-1})*	*^{1}H NMR (DMSO/TMS), δ*	*^{31}P NMR (ppm)*	*Mass-Spectrum: m/z, $[M]^+$*
2a	3300–3100, 2177, 1652[a]	3.42 (m, 4H, $2CH_2$), 6.56 (s, 1H, NH), 6.65 (s, 1H, NH), 7.39–7.92 (m, 5H, arom), 8.74 (s, 1H, NH)	–	228
2b	3450–3200, 2171, 1655[a]	2.37 (s, 3H, CH_3), 3.41 (m, 4H, $2CH_2$), 6.56 (s, 1H, NH), 6.61 (s, 1H, NH), 7.20–7.80 (m, 4H, $H_{arom.}$), 8.66 (s, 1H, NH)	–	242
2c	3540–3130, 2155, 1657[a]	3.36 (m, 4H, $2CH_2$), 6.66 (s, 1H, NH), 6.85 (s, 1H, NH), 7.52–7.93 (m, 4H, $H_{arom.}$), 8.92 (s, 1H, NH)	–	262
3a	3469, 3337, 3221, 3171, 1656, 1590[a]	3.43 (m, 2H, CH_2), 3.70 (m, 2H, CH_2), 6.30 (br s, 2H, NH_2), 6.86 (s, 1H, NH), 7.39–7.98 (m, 5H, arom), 8.77 (s, 1H, CONH), 10.20 (s, 1H, NH)	–	262
3b	3466, 3364, 3272, 3198, 1665, 1570[a]	2.35 (s, 3H, CH_3), 3.39 (m, 2H, CH_2), 3.66 (m, 2H, CH_2), 6.35 (br s, 2H, NH_2), 6.85 (s, 1H, NH), 7.23–7.88 (m, 4H, arom), 8.72 (s, 1H, CONH), 10.19 (s, 1H, NH)	–	276
3c	3416, 3315, 3258, 3190, 1643, 1578[a]	3.39 (m, 2H, CH_2), 3.66 (m, 2H, CH_2), 6.45 (br s, 2H, NH_2), 6.92 (s, 1H, NH), 7.50–7.99 (m, 4H, arom), 8.85 (s, 1H, CONH), 10.19 (s, 1H, NH)	–	296
4a	3058, 1617, 1541[a]	3.89 (m, 2H, CH_2), 4.15 (m, 2H, CH_2), 7.50–7.85 (m, 5H, arom), 10.29 (br s, 2H, SH + NH)	80.6	338
4b	3129, 1614,1542[a]	2.36 (s, 3H, CH_3), 3.87 (m, 2H, CH_2), 4.15 (m, 2H, CH_2), 7.58–7.84 (m, 4H, arom), 9.81 (br. s, 2H, SH + NH)	81.2	352
4c	3115, 1614, 1539[a]	3.90 (m, 2H, CH_2), 4.16 (m, 2H, CH_2), 7.60–7.86 (m, 4H, arom), 10.52 (br s, 2H, SH + NH)	80.1	372
5a	3331, 1622[a], 1530[a]	3.62–4.00 (m, 6H, $3CH_2$), 7.16–7.83 (m, 10H, arom), 9.67 (br s, 1H, NH)	60.1	428
5b	3329, 1618[a], 1534[a]	2.36 (s, 3H, CH_3), 3.30–3.95 (m, 6H, $3CH_2$), 7.15–7.72 (m, 9H, arom), 9.66 (br. s, 1H, NH)	59.3	442
6a	1613, 1530,1507	2.10 (d, 3H, CH_3), 3.63 (s, 3H, CH_3), 3.93 (m, 2H, CH_2), 4.07 (m, 2H, CH_2), 7.46–7.77 (m, 5H, arom)	65.6	366
6b	1613, 1511[a]	2.11 (d, 3H, CH_3), 2.34 (s, 3H, CH_3), 3.62 (s, 3H, CH_3), 3.92 (m, 2H, CH_2), 4.08 (m, 2H, CH_2), 7.27–7.66 (m, 4H, arom)	63.3	380
6c	1614, 1533, 1508	2.10 (d, 3H, CH_3), 3.62 (s, 3H, CH_3), 3.93 (m, 2H, CH_2), 4.07 (m, 2H, CH_2), 7.54–7.78 (m, 4H, arom)	62.7	400
6d	1592[a], 1508[a]	3.22–3.91 (m, 6H, $3CH_2$), 5.02–5.49 (dd, 2H, CH_2), 7.14–7.78 (m, 15H, arom)	60.1	518
6e	1596[a], 1515[a]	2.34 (s, 3H, CH_3), 3.24–3.92 (m, 6H, $3CH_2$), 5.11–5.44 (dd, 2H, CH_2), 7.15–7.64 (m, 14H, arom)	60.9	532
6f	1600[a], 1529[a]	3.24–3.94 (m, 6H, $3CH_2$), 5.07–5.43 (dd, 2H, CH_2), 7.15–7.80 (m, 14H, arom)	60.7	553
7a	1598[a], 1527[a]	3.22–3.56 (m, 2H, CH_2), 3.47 (s, 3H, CH_3), 3.70–4.03 (m, 4H, $2CH_2$), 7.14–7.84 (m, 10H, arom)	61.1	442
7b	1601[a], 1530[a]	2.38 (s, 3H, CH_3), 3.21–3.54 (m, 2H, CH_2), 3.47 (s, 3H, CH_3), 3.68–4.02 (m, 4H, $2CH_2$), 7.13–7.75 (m, 9H, arom)	61.2	456

[a]A band with a shoulder.



5	a	b	6	a	b	c	d	e	f
Ar	Ph	4-MeC_6H_4	**Ar**	Ph	4-MeC_6H_4	4-ClC_6H_4	Ph	4-MeC_6H_4	4-ClC_6H_4
Alk	—	—	**Alk**	Me	Me	Me	$PhCH_2$	$PhCH_2$	$PhCH_2$

1–4 **a** = Ph, **b** = 4-MeC_6H_4, **c** = 4-ClC_6H_4

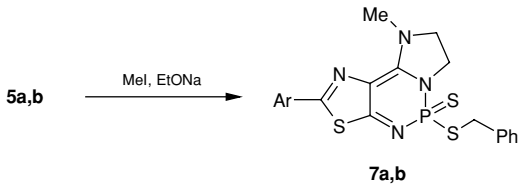
SCHEME 1

the expected integral intensity ratio. Moreover, the structure of products **2a–c** is unambiguously evidenced by the characterization data for their previously reported analogue, *N*-[cyano(imidazolidin-2-ylidene)methyl]acetamide [5].

On reacting with hydrogen sulfide in pyridine, nitriles **2a–c** were converted almost quantitatively to corresponding thioamides **3**, which were then treated with 2 equiv of P_2S_5 in pyridine by the known procedure [17–20] to furnish substituted derivatives **4** of 4,5,7,8-tetrahydroimidazo[1,2-*c*][1,3]thiazolo[4,5-*e*][1,3,2]diazaphosphinine, a novel-fused heterocyclic system. As found, compounds **4** are also obtainable directly from **2** by treating with phosphorus pentasulfide. However, the reaction course is complicated in this case, leading to much lower yields of products **4a–c** than in the conversion **2**→**3**→**4**.

By substituting two exchangeable hydrogen atoms in compounds **4a–c**, we obtained their benzyl and methyl derivatives **5–7**. With a moderately reactive alkylating agent such as benzyl chloride, it was possible to isolate both S-monosubstituted and N,S-disubstituted benzyl derivatives (compounds **5a,b** and **6d–f**, respectively) depending on the reagent ratio. At the same time, alkylation with a more reactive methyl iodide, even if used in an equimolar amount, provided exclusively *N*,*S*-dimethyl derivatives (compounds **6a–c**).

To prepare dialkyl derivatives **7a,b** analogous to **6** but containing different alkyl groups at the N and S atoms, we treated S-monosubstituted benzyl derivatives **5a,b** with methyl iodide.



7a **Ar** = Ph, **7b** **Ar** = 4-MeC_6H_4

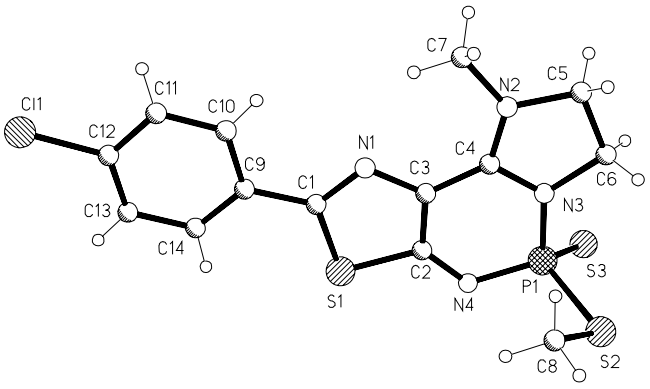


FIGURE 1 Perspective view and labeling scheme for molecule **6C**. Selected bond lengths (Å) and angles (degrees) for **6C**: P1–S2 2.0823(10), P1–S3 1.9276(10), N3–P1 1.706(2), N4–P1 1.618(2), C2–N4 1.326(3), C2–C3 1.403(3), C3–C4 1.407(3), C4–N3 1.361(3), C4–N2 1.322(3), C5–N2 1.454(3), C5–C6 1.523(4), C6–N3 1.462(3), C2–S1 1.750(3), C1–S1 1.764(2), C1–N1 1.293(3), C3–N1 1.377(3); N4–P1–N3 104.48(10), N4–P1–S3 119.95(9), N3–P1–S3 111.58(9), N4–P1–S2 108.63(10), N3–P1–S2 103.46(8), S3–P1–S2 107.51(5).

The identity of all compounds was verified by IR, NMR, and mass spectra, and **6c** was structurally determined by X-ray diffraction analysis, thus clearly indicating the presence of a new heterocyclic system, 4,5,7,8-tetrahydroimidazo[1,2-*c*][1,3]thiazolo[4,5-*e*][1,3,2]diazahosphinine.

The main geometrical parameters (bond lengths and angles) of the central tricyclic core of structure **6c** are shown in Fig. 1. The bond lengths of P1–S2 and P1–S3 are in the normal range for such bonds. The bond P1–N3 is longer than P1–N4, whereas the other structural parameters of the tricyclic core are typical of π-delocalized systems. The central core is planar (deviations of atoms from the least-squares plane do not exceed 0.021 Å), and the phenyl ring is practically coplanar with it (the dihedral angle S1–C1–C9–C14 is 1.07(9)°).

EXPERIMENTAL

NMR spectra were recorded on a Varian Gemini 300 spectrometer at 300 (^{1}H) and 80.95 MHz (^{31}P). ^{1}H NMR chemical shifts are given in δ (ppm) relative to Me_4Si as an internal standard. ^{31}P NMR spectra were recorded relative to 85% H_3PO_4 as an external standard. IR spectra were measured on a Specord M-80 spectrometer for KBr disks. Mass spectra were measured on a Surveyor MSQ instrument (Thermo Finnigan) using the atmospheric pressure chemical ionization (APCI) method and 0.1% aqueous solution of formic acid and acetonitrile as solvents.

N-[Cyano(imidazolidin-2-ylidene)methyl] carboxamides **2a–c**

To a suspension of **1a–c** (50 mmol) obtained by the known procedure [3,4] in propan-2-ol (100 mL), ethylenediamine (250 mmol) was added. The mixture was allowed to stand at room temperature (r.t.) for 5 min. The resulting precipitate was filtered off and washed with H_2O. The product was purified by recrystallization from EtOH.

N-(2-Amino-1-imidazolidin-2-ylidene-2-thioxoethyl) Carboxamides **3a–c**

To **2a–c** (4 mmol), pyridine (10 mL) and triethylamine (1 mL) were added, and dry hydrogen sulfide was bubbled through the mixture for 1.5–2 h (until a solution was formed). The mixture was allowed to stand at r.t. for 12 h. The resulting precipitate was filtered off and washed with EtOH. The product was purified by recrystallization from EtOH.

2-Aryl-5-mercapto-5-thioxo-4,5,7,8-tetrahydroimidazo[1,2-c][1,3]thiazolo[4,5-e][1,3,2]diazaphosphinine **4a–c**

A mixture of **3a–c** (2 mmol) and P_2S_5 (4 mmol) in anhydrous pyridine was refluxed for 4 h. Then it was treated with water (100 mL), filtered, washed with EtOH, and purified by recrystallization from DMF-H_2O (5:1) to give **4a–c**.

Compounds **4a–c** were also obtained by boiling **2a–c** (2 mmol) in pyridine (10 mL) with P_2S_5 (4 mmol) for 4 h. The products were isolated as described above. Yields were 35–42%. The samples of compounds **4a–c** obtained from **2** and **3** exhibited identical IR spectra.

2-Aryl-5-benzylthio-5-thioxo-4,5,7,8-tetrahydroimidazo[1,2-c][1,3]thiazolo[4,5-e][1,3,2]diazaphosphinine **5a,b**

To a suspension of **4a,b** (1 mmol) in anhydrous EtOH (5 mL), a solution of EtONa (1 mmol) in EtOH (5 mL) and a solution of benzyl chloride (1 mmol) in EtOH (5 mL) were added. The reaction mixture was heated under reflux for 1 min and then cooled. After filtering off NaCl, the solvent was evaporated in vacuo and the residue was treated with H_2O, filtered off, dried, and purified by recrystallization from DMF–EtOH (1:1).

9-Alkyl-5-alkylthio-2-aryl-5-thioxo-5,7,8,9-tetrahydroimidazo[1,2-c][1,3]thiazolo[4,5-e][1,3,2]diazaphosphinine **6a–f**

To a suspension of **4a–c** (1 mmol) in anhydrous EtOH (10 mL), a solution of EtONa (2 mmol) in EtOH (10 mL) and a solution of methyl iodide or benzyl chloride (2 mmol) in EtOH (10 mL) were added. The reaction mixture was heated under reflux for 1 min, cooled, and allowed to stand at r.t. for 12 h. The resulting precipitate was filtered off, washed with H_2O, dried, and purified by recrystallization from EtOH.

Compounds **6d,e** were also obtained by reacting **5a,b** (1 mmol) with benzyl chloride (1 mmol) and EtONa (1 mmol) in EtOH (20 mL). Yields were 88–92%. The samples of compounds **6d,e** obtained from **4a,b** and **5a,b** exhibited identical IR spectra.

2-Aryl-5-benzylthio-9-methyl-5-thioxo-5,7,8,9-tetrahydroimidazo[1,2-c][1,3]thiazolo[4,5-e][1,3,2]diazaphosphinine **7a,b**

To a solution of **5a,b** (1 mmol) in anhydrous EtOH (20 mL), a solution of EtONa (1 mmol) in EtOH (5 mL) and a solution of methyl iodide (1 mmol) in EtOH (5 mL) were added. The mixture was allowed to stand at room temperature for 12 h. The resulting precipitate was filtered off, washed with H_2O, dried in vacuo over P_2O_5, and purified by recrystallization from EtOH.

X-Ray Structure Determination for **6c**

Crystal Data. $C_{14}H_{14}ClN_4PS_3$, M 400.89, monoclinic, space group $P2_1/c$, a = 9.3448(4), b = 17.9691(7), c = 11.0539(4) Å, β = 112.558(2)°, V = 1714.13(12) Å^3, Z = 4, d_c = 1.553 g·cm^{-3}, μ = 0.684 mm^{-1}, $F(000)$ = 824, and crystal size ca. 0.12 × 0.25 × 0.43 mm.

Data Collection. All crystallographic measurements were performed at room temperature on a Bruker Smart Apex II diffractometer operating in the ω and φ scans mode. The intensities of 10,597 reflections were collected (3459 unique reflections, R_{merg} = 0.0296) in the range of 2.27 ≤ θ ≤ 26.36° using Mo K_α radiation (λ = 0.71078 Å). The multiscan absorption correction was applied (the ratio of minimum to maximum apparent transmission is 0.761124).

Structure Solution and Refinement. The structure was solved by direct methods and refined by the full-matrix least-squares technique in the anisotropic approximation for non-hydrogen atoms using the SHELXS97 and SHELXL97 programs [21,22]. All hydrogen atoms were placed at calculated positions and refined with a *riding model*. In the refinement, 3459 reflections including 2540 those with I ≥ 2σ(I) were used. Convergence was obtained at $R1$ = 0.0388, $wR2$ = 0.0983 for all reflections and $R1$ = 0.0613, $wR2$ = 0.1123, GOF = 1.003 for those observed (253 parameters; obs/variabl. 10.0; the largest and smallest peaks in the final difference map 0.29 and −0.33 e/Å^3). The weighting scheme is as follows: $\omega = 1/[\sigma^2(F_o^2) + (0.0602\ P)^2 + 0.5161\ P]$, where $P = (F_o^2 + 2F_c^2)/3$. Full crystallographic details have been deposited at the Cambridge Crystallographic Data Centre (CCDC). Any request to the CCDC for this material should quote the full literature citation and the reference number CCDC 768486.

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