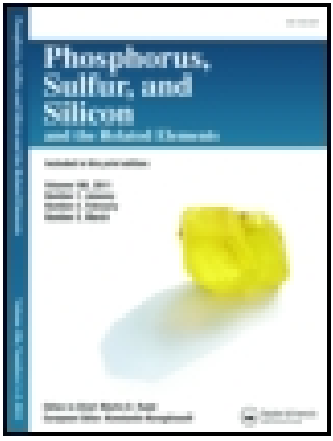

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Novel N,S- and S,S-Substituted Dienes Synthesized from Mercapto Triazole and Some Amine Derivatives

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Novel *N*, *S*- and *S*, *S*-Substituted Dienes Synthesized from Mercapto Triazole and Some Amine Derivatives

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*2-Nitrodiene **1** reacted with 3-mercapto-1,2,4-triazole and cyclohexyl thiol to yield compounds **3** and**4**, respectively. Compounds **7a–d** were obtained by reactions of **6a–d** with **2**. The novel N,S-substituted dienes **9a–c** were obtained by treatment of compound **6b** with the piperazine derivatives **8a–c**. Compound **6b** was reacted with **10** to give compound **11** as a new morpholine derivative. Compound **6b** gave a new monobutadienyl homopiperazine **13** when reacted with homopiperazine in methylene chloride. Compound **4** was structurally characterized by single-crystal X-ray diffraction.*

Keywords Mono(thio)substituted nitrodiene; 2-nitrohalodiene; 3-mercapto-1,2,4-triazole; morpholine; piperazine; *N*, *S*-substituted nitrodiene

INTRODUCTION

The synthesis of unsaturated thio-substituted halogenated organic compounds from the reaction of various halogenated alkenes and dienes with a number of thiols were published.[1–8]It is known that *N*, *S*–substituted butadienes were prepared from the reaction of mono(thio)substituted compounds with amines (primary amines, piperazine, morpholine, piperidine, etc).[9–16] There are many studies about the reactions of some *N*-nucleophiles, such as benzotriazol, 3,5-dimethylpyrazole, and indol with nitrodienes.[17–19] Triazoles, and in particular the 1,2,4-triazole ring, have been incorporated into a wide variety of therapeutically interesting drug candidates, including anti-inflammatory, sedatives, antianxiety, and antimicrobial agents.[20–28] Some piperazine and piperidine compounds have also been subject to

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medicinal applications and gen transfer studies due to their interesting biological activity and chemical effects.[29–31]

In this article, we synthesized and characterized new *N*, *S*-and *S*, *S*-substituted nitrobutadienes. Reaction of mono(thio)substituted diene compounds with some amines and triazoles provided the corresponding *N*, *S*-substituted halodiene derivatives.

RESULTS AND DISCUSSION

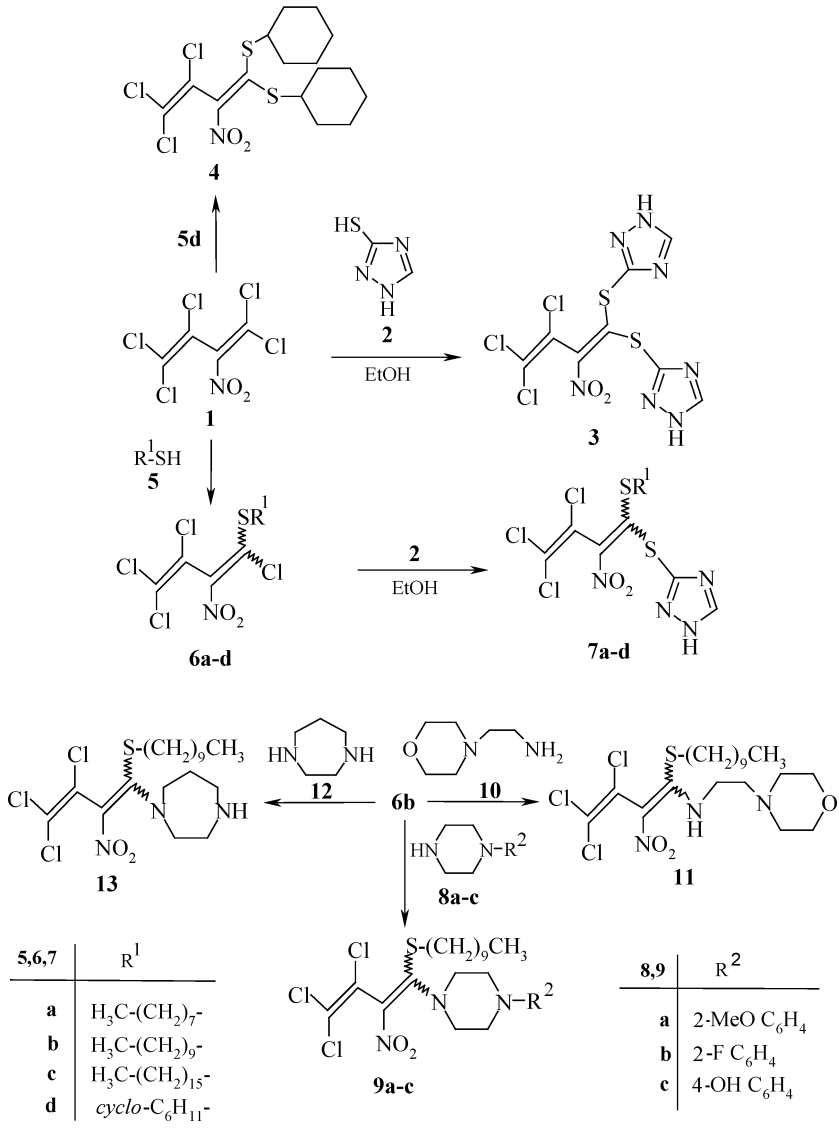
The position adjacent to $=C(NO_2)$- is the most favorable for nucleophilic attack in the halonitrovinyl moiety. Polychlorinated nitrodienes are highly electrophilic compounds. The quantum-chemical analysis shows that halonitrodienes must be active in processes of nucleophilic vinylic substitution.[32–38]

Reaction of **1** with the thiols **2** and **5d** resulted in the formation of the new butadienes **3** and **4**. The *S*, *S*-disubstituted nitrobutadiene **4** was obtained as a by-product in the synthesis of **6d** using the standard work up procedure (see Experimental Section). The mono(thio)substituted dienes **6a–d**[29,13,39,7] react with 3-mercapto-1,2,4-triazole to give the new derivatives **7a–d**. Compound **6b** reacts with the piperazines **8a–c** to give the *N*, *S*-substituted butadienes **9a–c**. Treatment of **6b** with the amines **10** and **12** results in the formation of the substitution products **11** and **13**, respectively (Scheme 1).

The structures of the new compounds were established by their spectroscopic data. In the IR spectra the >NH groups of the triazole rings[40] showed characteristic bands in the range of 3100-3300 cm^{-1}. In the case of compounds **11** and **13**, characteristic NH bands at 3400 and 3450 cm^{-1} were observed. The presence of the OH-group in compound **9c** is revealed by a characteristic band at 3350 cm^{-1}.

The stereochemistry of the butadiene **4** was confirmed by the result of a single crystal X-ray structure determination. Experimental details for data collection and structure refinement are summarized in Table I. An ORTEP diagram of the molecular structure of **4** in the crystal with atom numbering scheme is shown in Figure 1. Selected bond lengths and angles can be found in Table II. Physical properties and spectroscopic data of the synthesized compounds are listed in Table III and Table IV.

The C-C bond lengths of the butadiene chain in **4** are 1.332(8), 1.482(9), and 1.343(6) Å for C(2)-C(1), C(3)-C(2), and C(4)-C(3), respectively. The bond angles C(1)-C(2)-C(3) and C(2)-C(3)-C(4) are 123.3(6)° and 126.0(4)°, respectively. The diene system is not planar. The torsion angle C(4)-C(3)-C(2)-C(1) is 106.3(7)°.



SCHEME 1

EXPERIMENTAL

Melting points were determined on a Buchi B-540 capillary apparatus and were uncorrected. Elemental analyses were carried out on a Carlo Erba 1110 analyzer. Infrared spectra were recorded on a Shimadzu

TABLE I Crystallographic Data and Structure Refinement for Compound 4

Sum formula	$(C_{16}H_{22}NO_2Cl_3S_2)$
f_w (g.mol^{-1})	430.83
Crystal dimensions (mm)	0.60 × 0.40 × 0.20
Crystal system	monoclinic
Space group	$P2_1/n$
Lattice parameters	
a (Å)	12.0862(12)
b (Å)	11.1625(8)
c (Å)	16.337(1)
α (°)	90
β (°)	110.840(4)
γ (°)	90
Vol [Å^3]	2059.9(3)
Z	4
D_{calc}(g.cm^{-3})	1.338
μ [cm^{-1}]	6.49
F(000)	864.00
Index ranges	$-14 \leq h \leq 14$ $-13 \leq k \leq 13$ $-19 \leq l \leq 19$
Reflections collected	66140
Independent reflections	3579[R_{int}= 0.059]
Data/restraints/parameters	1717/0/239
Goodness-of-fit on F^2	0.752
Final R indices [I>3 σ (I)]	$R = 0.074$, $wR = 0.015$
Largest diff. peak and hole	0.40 and −0.41 e. Å^{-3}

$R = \Sigma \; ||Fo| - |Fc||/\Sigma \; |Fo|$, $R_w = [\Sigma \; w \; (|Fo| - |Fc|)2/\Sigma w \; Fo2]^{1/2}$

FTIR-8101 spectrometer in KBr discs. ^{1}H and ^{13}C NMR spectra were measured on Varian^{UNITY} INOVA spectrometer. Mass spectra were obtained using Finnigan LCQ Advantage Max. LC/MS. Thin-layer chromatography was performed on 0.5 mm × 20 cm × 20 cm E. Merck silica gel plates (60 F-254). Column chromatography was conducted over Silica gel (63–200 μm), available from E. Merck. All chemicals were reagent grade and used without further purification. Moisture was excluded from the glass apparatus using $CaCl_2$ drying tubes.

Preparation of Compounds 4 and 6d. General Procedure

To 2 g of 1,1,3,4,4-pentachloro-2-nitro-1,3-butadiene **1** was added an equimolar amount of the thiol **5d** and the mixture was vigorously stirred without solvent at room temperature until completion of the reaction. Stirring of the mixture was continued for 36 h. Then, chloroform was

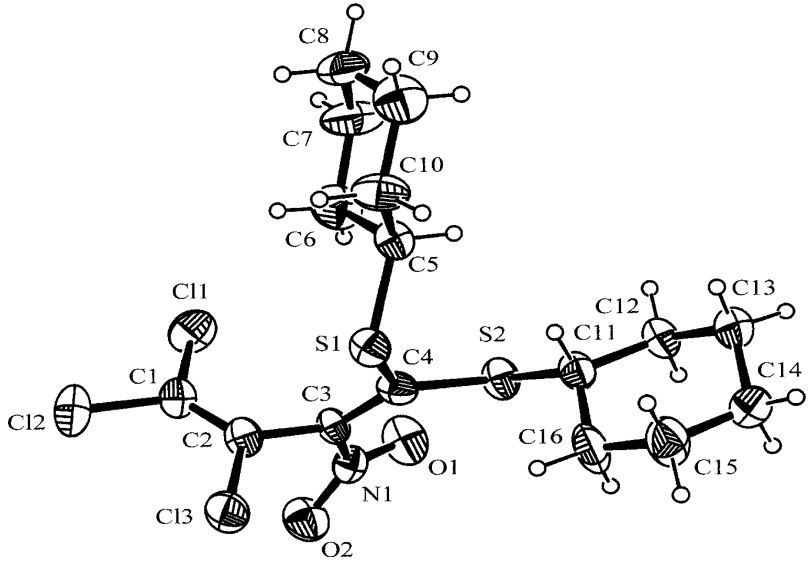


FIGURE 1 ORTEP view of the molecular structure of **4** in the crystal; displacement ellipsoids are drawn at the 30% probability level.

added to the reaction mixture. The organic layer was separated, washed with water several times, and dried over anhydrous $CaCl_2$ or $MgSO_4$. After removal of the solvent, a mixture containing the compounds **4** and **6d** was obtained. The pure compounds **4** and **6d** were seperated by column chromatography over silica gel using petroleum ether as eluent.

Preparation of Compounds 3 and 7a–d, General Procedure

To a solution of 0.5 g of **1** or **6a–d** in 10 mL of ethanol was added an equimolar amount of **2** in 20 mL of ethanol and the reaction mixture was vigorously stirred at room temperature until completion of the reaction. Stirring of the reaction mixture was continued for 4 h. Then, chloroform was added to the reaction mixture. The organic layer was separated and washed with water several times and dried over anhydrous $CaCl_2$ or $MgSO_4$. After removal of the solvent, the products were either crystallized or purified by column chromatography over silica gel.

Preparation of Compounds 9a–c, 11 and 13, General Procedure

To a solution of 0.2 g of **6b** in 10 mL of dichloromethane was added an equimolar amount of the respective amine in 20 mL of dichloromethane

TABLE II Selected Bond Lengths [Å] and Angles [°] for Compound 4

Cl(3)	C(2)		1.723(7)	Cl(1)	C(1)		1.699(7)
Cl(2)	C(1)		1.711(6)	S(2)	C(4)		1.739(6)
S(2)	C(11)		1.831(6)	S(1)	C(4)		1.776(5)
S(1)	C(5)		1.827(6)	O(1)	N(1)		1.214(9)
O(2)	N(1)		1.230(7)	N(1)	C(3)		1.454(6)
C(4)	C(3)		1.343(6)	C(3)	C(2)		1.482(9)
C(1)	C(2)		1.332(8)	C(5)	C(10)		1.540(7)
C(5)	C(6)		1.490(1)	C(12)	C(11)		1.520(1)
C(12)	C(13)		1.510(1)	C(10)	C(9)		1.540(1)
C(15)	C(16)		1.570(1)	C(15)	C(14)		1.490(1)
C(16)	C(11)		1.470(1)	C(7)	C(6)		1.520(1)
C(7)	C(8)		1.520(1)	C(14)	C(13)		1.490(1)
C(9)	C(8)		1.480(1)				
C(4)	S(2)	C(11)	106.1(3)	C(4)	S(1)	C(5)	100.4(2)
C(3)	N(1)	O(1)	119.3(4)	C(3)	N(1)	O(2)	117.4(5)
O(1)	N(1)	O(2)	123.3(5)	C(3)	C(4)	S(2)	124.0(4)
C(3)	C(4)	S(1)	115.3(4)	S(2)	C(4)	S(1)	120.7(3)
C(2)	C(3)	N(1)	113.5(4)	C(2)	C(3)	C(4)	126.0(4)
N(1)	C(3)	C(4)	120.3(5)	C(2)	C(1)	Cl(1)	122.1(5)
C(2)	C(1)	Cl(2)	122.6(6)	Cl(1)	C(1)	Cl(2)	115.3(3)
Cl(3)	C(2)	C(3)	115.5(4)	Cl(3)	C(2)	C(1)	121.1(5)
C(3)	C(2)	C(1)	123.3(6)	C(10)	C(5)	C(6)	110.6(5)
C(10)	C(5)	S(1)	106.4(4)	C(6)	C(5)	S(1)	112.2(4)
C(11)	C(12)	C(13)	110.3(6)	C(9)	C(10)	C(5)	109.3(6)
C(16)	C(15)	C(14)	109.2(9)	C(11)	C(16)	C(15)	111.4(6)
C(6)	C(7)	C(8)	111.7(9)	C(13)	C(14)	C(15)	113.5(7)
S(2)	C(11)	C(12)	105.7(5)	S(2)	C(11)	C(16)	113.4(4)
C(12)	C(11)	C(16)	112.4(6)	C(8)	C(9)	C(10)	111.5(6)
C(5)	C(6)	C(7)	111.5(6)	C(7)	C(8)	C(9)	111.9(7)
C(12)	C(13)	C(14)	110.8(6)				

and the reaction mixture was vigorously stirred at room temperature until completion of the reaction. Stirring of the reaction mixture was continued for 4 h. Then, chloroform was added to the reaction mixture. The organic layer was separated, washed with water several times, and dried over anhydrous $CaCl_2$ or $MgSO_4$. After removal of the solvent, the products were either crystallized or purified by column chromatography over silica gel.

X-Ray Diffraction

Suitable single crystals of **4** were obtained by slow evaporation of a chloroform solution. A single crystal of **4** was mounted on an Rigaku R-Axis Rapid-S diffractometer equipped with a graphite monochromatized

TABLE III Analytical and IR Spectroscopic Data of the New Compounds

	Mol. formula (mol. wt.)	Yield (%)	m.p (°C)	Elemental Analysis calcd/found C	H	N	R_f (Solvent)	MS m/z M^+	IR (cm^{-1})
3	$C_8H_4S_2N_7Cl_3O_2$ (400.65)	48	171–172	23.98 23.37	1.01 1.50	24.47 23.71	0.16 (EtAc)	401	2700, 2750, 2850, 2950 (C—H), 1600 (C=C), 1240, 1540 (C—NO_2), 3200 (NH)
4	$C_{16}H_{22}NO_2Cl_3S_2$ (430.84)	28	109–110	44.60 44.56	5.15 5.10	3.25 3.43	0.13 (Pet. ether)	—	2800, 2900 (C—H), 1600 (C=C), 1270, 1530 (C—NO2)
7a	$C_{14}H_{19}S_2N_4Cl_3O_2$ (445.82)	39	94–95	37.72 36.83	4.30 3.79	12.57 12.96	0.83 (EtAc).	446	2870, 2940 (C—H), 1610 (C=C), 1250, 1260, 1530 (C—NO2), 3300 (NH)
7b	$C_{16}H_{23}S_2N_4Cl_3O_2$ (473.87)	33	Oil	40.55 40.46	4.89 4.54	11.82 11.38	0.86 ($CHCl_3$)	472	2800, 2900 (C—H), 1560 (C=C), 1230, 1540 (C—NO2), 3195 (NH)
7c	$C_{22}H_{35}S_2N_4Cl_3O_2$ (558.03)	35	80–81	47.35 47.43	6.32 5.83	10.04 9.46	0.90 (EtAc)	557	2750, 2840, 2850 (C—H), 1600 (C=C), 1240, 1270, 1530 (C—NO2), 3100 (NH)
7d	$C_{12}H_{13}S_2N_4Cl_3O_2$ (415.75)	58	147–148	34.67 34.86	3.15 3.20	13.48 13.28	0.76 (EtAc)	415	2850, 2950 (C—H), 1600 (C=C), 1260, 1540 (C—NO2), 3260 (NH)
9a	$C_{25}H_{36}SN_3Cl_3O_3$ (565.00)	45	Oil	53.15 52.55	6.42 6.84	7.44 7.40	0.17 ($CHCl_3$)	—	2800, 2900 (C—H), 1595 (C=C), 1250, 1530 (C—NO2)
9b	$C_{24}H_{33}SN_3Cl_3O_2F$ (552.96)	54	72–73	52.13 52.42	6.02 6.33	7.60 7.50	0.72 (EtAc/CCI_4 1:4)	—	2800, 2900, 2950 (C—H), 1610 (C=C), 1270, 1535 (C—NO2)
9c	$C_{24}H_{34}SN_3Cl_3O_3$ (550.97)	52	106–107	52.32 52.18	6.22 5.94	7.63 7.69	0.71 (EtAc)	—	2750, 2850 (C—H), 1595 (C=C), 1275, 1530, 1540 (C—NO2), 3350 (C—OH)
11	$C_{20}H_{34}SN_3Cl_3O_3$ (502.93)	59	Oil	47.76 47.98	6.81 6.75	8.36 7.84	0.52 (MeOH)	—	2850, 2900 (C—H), 1600 (C=C), 1240, 1540 (C—NO_2), 3400 (C—NH)
13	$C_{19}H_{32}SN_3Cl_3O_2$ (473.90)	39	Oil	48.26 47.95	6.82 6.60	8.89 9.07	0.28 (MeOH)	473	2850, 2950 (C—H), 1630 (C=C), 1230, 1530 (C—NO_2), 3450 (C—NH)

TABLE IV ^{1}H and ^{13}C NMR Data of the New Compounds

		^{1}H/^{13}C-NMR δ (ppm)
3	^{1}H (DMSO-d_6)	8.57 (s, 2H, CH), 8.67 (s, 2H, NH)
4	^{1}H ($CDCl_3$)	1.18–2.01 (m, 20H, CH_2), 3.24–3.47 (m, 2H, SCH)
	^{13}C ($CDCl_3$)	25.5, 25.6, 26.3, 33.8, 49.5, 51.3, 123.2, 128.0, 142.9, 157.1
7a	^{1}H (DMSO-d_6)	0.71–0.97 (m, 3H, CH_3), 1.12–1.80 (m, 12H, $(CH_2)_6$), 2.51–3.03 (m, 2H, SCH_2), 8.74 (s, 1H, CH), 8.80 (s, 1H, NH)
7b	^{1}H (DMSO-d_6)	0.73–0.98 (m, 3H, CH_3), 0.98–2.09 (m, 16H, $(CH_2)_8$), 2.50–2.68 (m, 2H, SCH_2), 8.55 (s, 1H, CH), 9.53 (s, 1H, NH)
7c	^{1}H (DMSO-d_6)	0.84–1.24 (m, 3H, CH_3), 1.26–1.60 (m, 28H, $(CH_2)_{14}$), 2.92–3.32 (m, 2H, SCH_2), 8.75 (s, 1H, CH), 8.81 (s, 1H, NH)
7d	^{1}H (DMSO-d_6)	1.20–2.50 (m, 11H, C_6H_{11}), 8.77 (s, 1H, CH), 8.83 (s, 1H, NH)
9a	^{1}H ($CDCl_3$)	0.82–0.90 (m, 3H, CH_3), 1.24–1.67 (m, 16H, $(CH_2)_8$), 1.69–1.78 (m, 3H, OCH_3), 2.91–3.05 (m, 2H, SCH_2), 3.26–3.91 (m, 8H, NCH_2), 6.89–7.24 (m, 4H, arom–H)
	^{13}C ($CDCl_3$)	9.6, 18.1, 24.2, 24.5, 24.7, 24.9, 25.0, 25.3, 25.5, 27.3, 31.0, 31.6, 33.0, 45.9, 48.8, 51.1, 107.3, 113.6, 116.7, 120.1, 122.4, 147.8
9b	^{1}H ($CDCl_3$)	0.81 (t, $J = 6.8$ Hz, 3H, CH_3), 1.19–1.73 (m, 16H, $(CH_2)_8$), 2.91 (t, $J = 7.3$ Hz, 2H, SCH_2), 3.17–3.75 (m, 8H, NCH_2) 6.85–7.19 (m, 4H, arom-H)
	^{13}C ($CDCl_3$)	14.3, 22.9, 28.9, 29.5, 29.6, 29.7, 30.0, 32.1, 35.8, 39.5, 44.0, 47.8, 50.6, 53.6, 116.7, 119.6, 119.8, 124.4, 125.0, 127.1, 138.8, 155.0, 157.0, 169.7
9c	^{1}H ($CDCl_3$)	0.86 (t, $J = 6.8$ Hz, 3H, CH_3), 1.24–1.68 (m, 16H, $(CH_2)_8$), 2.95 (t, $J = 7.3$ Hz, 2H, SCH_2), 3.17–3.78 (m, 8H, NCH_2), 6.76 (d, $J = 8.8$ Hz, 2H, arom-H), 6.85 (d, $J = 8.8$ Hz, 2H, arom-H)
	^{13}C ($CDCl_3$)	14.3, 22.9, 28.9, 29.3, 29.5, 29.6, 29.7, 30.0, 32.1, 35.8, 51.3, 53.5, 116.4, 118.5, 119.6, 123.8, 125.0, 127.0
11	^{1}H ($CDCl_3$)	0.85 (t, $J = 6.8$ Hz, 3H, CH_3), 1.24–1.67 (m, 20H, $(CH_2)_8$ and NCH_2), 2.62–2.67 (m, 2H, SCH_2), 2.89–4.07 (m, 8H, morpholine-CH_2)
13	^{1}H ($CDCl_3$)	0.81–1.08 (m, 3H, CH_3), 1.12–1.76 (m, 16H, $(CH_2)_8$), 2.42–2.74 (m, 2H, SCH_2), 2.81–4.27 (m, 10H, CH_2)
	^{13}C ($CDCl_3$)	14.3, 22.9, 24.7, 28.2, 29.6, 32.1, 36.6, 39.5, 46.2, 50.8, 54.8, 66.9, 70.5, 118.7, 125.4, 126.7, 162.2, 171.9

MoK_α radiation source ($\lambda = 0.71073$ Å). The structure was solved by SIR 92 [41] and refined with CRYSTALS.[42] The positions of the H atoms bonded to C atoms were calculated (C—H distance 0.96 Å), and refined using a riding model. The H atom displacement parameters were restricted to be $1.2U_{eq}$ of the parent atom. All calculations were performed using the Crystal Structure Analysis software package.[43] An ORTEP-III view of the molecular structure of **4** is given in Figure 1.[44] Crystallographic data (excluding structure factors) for the structure reported in

this article have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication No. CCDC 278799.[45]

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