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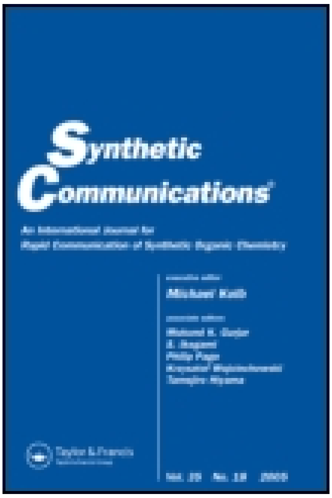

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Cis-1,4-bis(triphenylphosphonium)-2-butene Peroxodisulfate as an Efficient Reagent for the Synthesis of Phenacyl Thiocyanates and Phenacyl Azides

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CIS-1,4-BIS(TRIPHENYLPHOSPHONIUM)-2-BUTENE PEROXODISULFATE AS AN EFFICIENT REAGENT FOR THE SYNTHESIS OF PHENACYL THIOCYANATES AND PHENACYL AZIDES

Rashid Badri[1] **and Maryam Gorjizadeh**[2]
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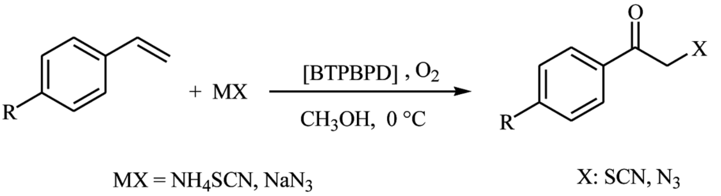
[2]*Department of Chemistry, Islamic Azad University, Shoushtar Branch, Shoushtar, Iran*

GRAPHICAL ABSTRACT



Abstract *Styrenes efficiently undergo thiocyanation and azidation with cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate at 0°C to furnish the corresponding thiocyanatoketones and azidoketones. This method is a direct, one-pot synthesis under mild condition using methanol as solvent.*

Keywords Cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate; phenacyl azide; phenacyl thiocyanate; styrene

INTRODUCTION

The phenacyl compounds are useful intermediates in organic synthesis and have attracted a great deal of interest.[1] Azide and thiocyanate derivatives of α-keto compounds opened an important area of heterocyclic chemistry because many of them are subunits of natural products and pharmaceutical agents. Phenacyl thiocyanates and phenacyl azides have been conventionally prepared from α-haloketones via nucleophilic displacement with thiocyanate or azide. These methods, however, often require drastic reaction conditions leading to poor yields of products. Recently, some methods for the synthesis of azide and thiocyanate derivatives of α-keto

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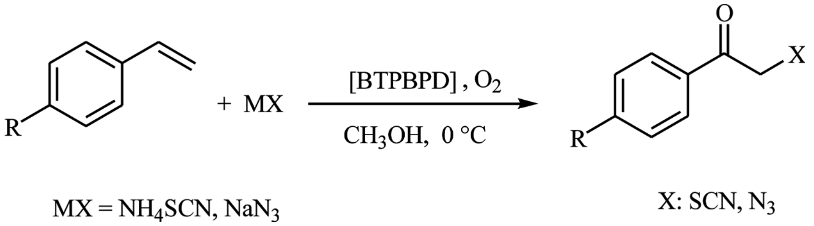


Scheme 1. Synthesis of phenacyl thiocyanates and phenacyl azides from styrene and 1,4-bis-(triphenylphosphonium)-2-butene peroxodisulfate as an oxidant.

compounds by using cerium(IV) ammonium nitrate,[2] iodine/MeOH,[3] Me_3SiN_3-CrO_3,[4] and phenacyl bromides in hydroalcoholic media[5] were reported. Peroxodisulfate ion is an excellent and versatile oxidant, used mostly for the oxidation of compounds in aqueous solution.[6] In spite of the great convenience of using $K_2S_2O_8$, $Na_2S_2O_8$, or $(NH_4)_2S_2O_8$ and relatively high oxidation potential, many oxidation reactions by peroxodisulfate do not proceed at a convenient rate. This can be largely attributed to the rate-limiting homolysis, which has activation energy of approximately 30 Kcal/mol. Thus, certain limitations may be observed with these reagents. The decomposition of the peroxodisulfate ion requires strong mineral acids and heavy metal ions[7] as catalysts and also protic and polar solvents are needed. Recently, the modification of $K_2S_2O_8$, $Na_2S_2O_8$, or $(NH_4)_2S_2O_8$ has attracted a great deal of attention.[8]

Recently, cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate (BTPBPD), an inexpensive and environmentally safe oxidation reagent, has been used for synthesis of 2,5-disubstituted-1,3,4-oxadiazoles,[9] iodination of aromatic compounds,[10] and synthesis of β-nitrato alcohols.[11] Herein, we report preparation of phenacyl thiocyanates and phenacyl azides directly from alkenes with (BTPBPD) as an efficient oxidation reagent (Scheme 1).

RESULTS AND DISCUSSION

Cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate can be readily prepared by adding an aqueous solution of potassium peroxodisulfate to a solution of cis-1,4-bis(triphenylphosphonium)-2-butene dichloride in water. It is a very stable white solid that can be stored for months without losing its activity.[11]

We used 4-methoxy styrene as a model compound and reacted it with cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate as an oxidant in different solvents. Thin-layer chromatographic (TLC) analysis showed that the oxidant acted very efficiently in methanol, and 2 mmol of the BTPBPD is enough to convert the 1 mmol of substrate to its corresponding 4′-methoxy-2-thiocyanatoacetophenone in good isolated yield. At room temperature, thiocyanation with oxidant in methanol gave 1,2-dithiocyanate and phenacyl thiocyanate in a 1:2 ratio. This reaction was also tested under 0 °C condition; the results clearly showed that the reaction completed in 1 h and was associated with decreasing in 1,2-dithiocyanate, whereas the main product of the reaction was phenacyl thiocyanate (Table 1). With the

Table 1. Consideration of different conditions for thiocyanation of 4-methoxystyrene

No.	Solvent	BTPBPD (mmol)	Temperature	O_2 atmosphere	Time (min)	Conversion (%)[a]	Yields (%) Phenacyl thiocyanate	Yields (%) 1,2-Dithiocyanate
1	Et_2O	2	Rt	No	120	50	5	30
2	CH_2Cl_2	2	Rt	No	120	60	10	40
3	$CHCl_3$	2	Rt	No	120	70	10	45
4	CH_3CN	2	Rt	No	80	100	15	70
5	CH_3OH	2	Rt	No	60	100	30	60
6	CH_3OH	1.5	Rt	No	60	70	15	40
7	CH_3OH	2	0 °C	No	60	100	50	40
8	CH_3OH	2	0 °C	Yes	60	100	90	—

[a]According to GC analysis.

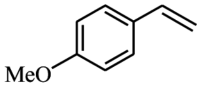
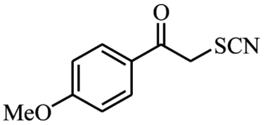
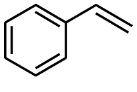
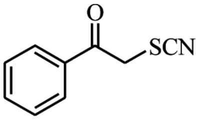
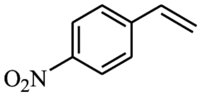
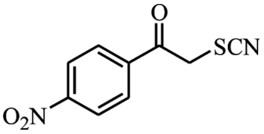
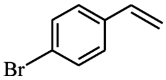
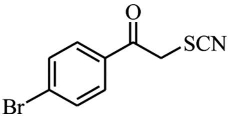
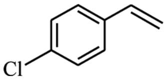
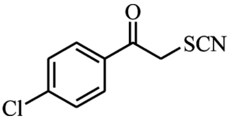
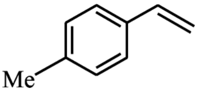
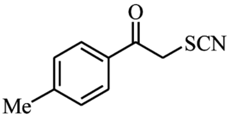
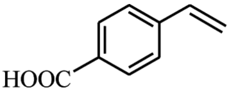
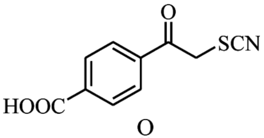
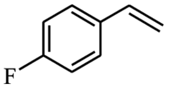
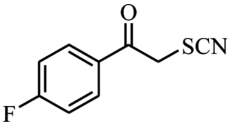
reasonable assumption that oxygen takes part in the reaction leading to the phenacyl thiocyanate, the reaction of 4-methoxystyrene with BTPBPD and ammonium thiocyanate was conducted in an atmosphere of oxygen. In this case the phenacyl thiocyanate was formed in 90% yield and no dithiocyanate was observed. Table 2 shows the scope and limitations of these reactions using several styrene derivatives. The formation of products can be easily detected by the characteristic ^{13}C signal of the ^{-}SCN group at ~112 ppm.[12] Both styrene derivatives with electron-withdrawing group and electron-donating groups provided the corresponding α-thiocyanatoketones in good yields. To know the efficiency of this procedure, we tested the effectiveness of the other oxidant compounds by replacing BTPBPD with $K_2S_2O_8$, $(NH_4)_2S_2O_8$, and saturated oxidant at the identical reaction conditions. We have found that the formation of α-thiocyanatoketones occurred rather slowly, and yields of products were reduced to 5–10% on average compared to the results obtained using BTPBPD. This may be due to the low solubility of these reagents in reaction medium. However, no reaction was observed in the absence of oxidant, even over a long reaction time (12 h). It is noteworthy that with all substrates used in this procedure, no evidence for the formation of 1,2-dithiocyanates as by-product of the reactions was observed.

Prompted by the success of this reaction, we decided to explore the possibility of synthesizing phenacyl azides using a similar strategy. The reactions worked well to give the corresponding azidoketones with equal efficiencies in the cases of thiocyanation reactions (Table 3). ^{13}C resonance of α-carbon in phenacyl azide that shift from ~115 ppm in styrene to ~55 ppm is very characteristic for azide functionality.[12]

A plausible reaction pathway for the present reaction is shown in Scheme 2. Sulfate anion radical gained an electron from thiocyanate or azide anion and oxidized the anion to a radical, which then reacted with styrene to form the benzylic radical. This can trap oxygen to form the peroxyradical, which eventually oxidized by cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate to form the products.

In summary, we have demonstrated that thiocyanatoketones and azidoketones can be generated directly from styrenes in the presence of BTPBPD. The notable features of this procedure are good conversions, simplicity in operation, cleaner reaction profiles, and ready availability of reagents at low cost, which make it a

Table 2. Conversion of different styrenes to phenacyl thiocyanates using BTPBPD

Compound	Substrate	Product[a]	Time (min)	Yield (%)
1			50	85
2			65	80
3			80	70
4			65	92
5			80	80
6			60	89
7			80	65
8			80	70

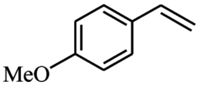
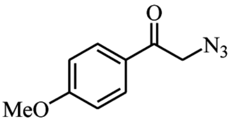
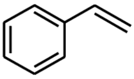
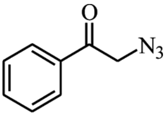
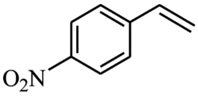
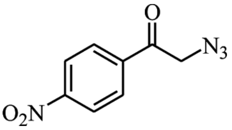
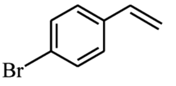
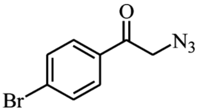
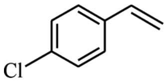
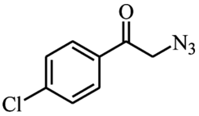
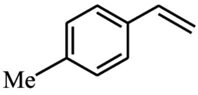
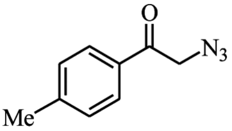
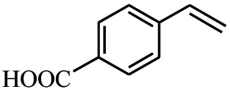
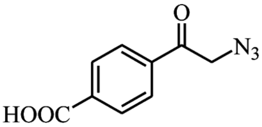
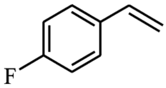
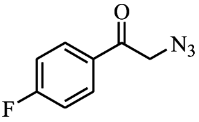
[a] Products were identified by comparison of their physical and spectral data with those of authentic samples.

useful and attractive strategy for the synthesis of phenacyl compounds of synthetic importance.

EXPERIMENTAL

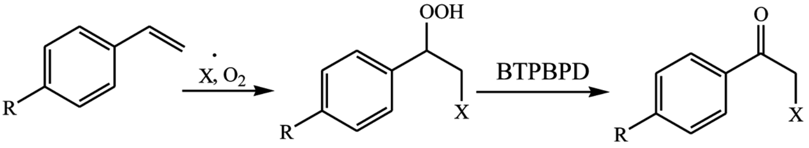
All products were characterized by comparison of their physical data, infrared (IR), ^{1}H NMR, and ^{13}C NMR spectra with authentic samples.[13,2] The IR spectra were recorded on a Bomem FT-IR spectrometer. ^{1}H NMR and ^{13}C NMR spectra were taken on a 400-MHz Bruker spectrometer. The C, H, P, N, S, F, and Br analyses were performed by the microanalytical service of the Research Institute

Table 3. Conversion of different styrenes to phenacyl thiocyanates using BTPBPD

Compound	Substrate	Product[a]	Time (min)	Yield (%)
9			50	95
10			65	85
11			80	70
12			65	92
13			80	80
14			60	89
15			80	73
16			90	70

[a] Products were identified by comparison of their physical and spectral data with those of authentic samples.

$$X^{-} \xrightarrow{\text{BTPBPD}} X^{\bullet} \qquad X = N_3, \text{SCN}$$



Scheme 2.

of the Petroleum Industry (NIOC). Cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate was prepared, and other chemicals were purchased from Merck (Darmstadt, Germany). All of the produts reported here were pure as judged by TLC, FTIR, and NMR analysis of the reaction mixture.

Preparation of Cis-1,4-bis(triphenylphosphonium)-2-butene Peroxodisulfate

The oxidant was prepared as described in our previous articles.[11]

General Procedure for the Preparation of Phenacyl Thiocyanate

A stock solution was prepared by dissolving 1 mmol of styrene and 1.5 mmol NH_4SCN in CH_3OH saturated with oxygen. Cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate (2 mmol) was also dissolved in methanol saturated with oxygen and then was added in small portions at 0 °C to a stock solution. The progress of the reaction was monitored by TLC. On completion of reaction, the reaction mixture was filtered. The solvent was evaporated; water (20 mL) was added and extracted with diethyl ether (3 × 15 mL). The combined organic layers were concentrated in vacuo; the resulting product was directly charged on a small silica-gel column and eluted with a mixture of ethyl acetate and n-hexane (1:4) to afford the pure product.

General Procedure for the Preparation of Phenacyl Azide

A stock solution was prepared by dissolving 1 mmol of styrene and 1.5 mmol sodium azide in CH_3OH saturated with oxygen. Cis-1,4-bis(triphenylphosphonium)-2-butene peroxodisulfate (2 mmol) was also dissolved in methanol saturated with oxygen and then was added in small portions at 0 °C to a stock solution. The progress of the reaction was monitored by TLC. On completion of reaction, the reaction mixture was filtered. The solvent was evaporated; water (20 mL) was added and extracted with diethyl ether (3 × 15 mL). The combined organic layers were concentrated in vacuo; the resulting product was directly charged on a small silica-gel column and eluted with a mixture of ethyl acetate and n-hexane (1:4) to afford the pure product.

Representative Examples of Spectra Data of Phenacyl Thiocyanate

Compound 2. Yield (0.142 g, 80%); IR: 2152 (SCN), 1689 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.61$ (s, 2H), 7.42–7.86 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 40.6$ (CH_2), 111.2 (SCN), 128.2 (2 × CH), 130.0 (2 × CH), 130.3 (CH), 134.1 (C), 185.1 (CO). Anal. calcd. for C_9H_7NOS: C, 60.99; H, 3.98; N, 7.90; S, 18.09. Found: C, 60.87; H, 3.97; N, 7.99; S, 18.22.

Compound 3. Yield (0.155 g, 70%); IR: 2153 (SCN), 1681 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.51$ (s, 2H), 7.91 (d, 2H), 8.50 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 38.2$ (CH_2), 111.3 (SCN), 123.2 (2 × CH), 134.0 (2 × CH), 139.3 (C), 148.1 (C), 185.1 (CO). Anal. calcd. for $C_9H_6N_2O_3S$: C, 48.64; H, 2.72; N, 12.60; S, 14.43. Found: C, 48.67; H, 2.78; N, 12.59; S, 14.22.

Compound 4. Yield (0.235 g, 92%); IR: 2151 (SCN), 1686 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.60$ (s, 2H), 7.41 (d, 2H), 7.88 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 40.7$ (CH_2), 111.5 (SCN), 127.9 (C), 132.3 ($2 \times CH$), 133.1 (C), 134.2 ($2 \times CH$), 185.1 (CO). Anal. calcd. for C_9H_6NOSBr: C, 42.21; H, 2.36; N, 5.47; S, 12.52; Br, 31.20. Found: C, 42.27; H, 2.38; N, 5.49; S, 12.62; Br, 31.35.

Compound 6. Yield (0.170 g, 89%); IR: 2150 (SCN), 1680 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 2.90$ (s, 3H), 4.62 (s, 2H), 7.34 (d, 2H) 7.58 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 22.7$ (CH_3), 41.2 (CH_2), 111.5 (SCN), 131.4 ($2 \times CH$), 132.3 ($2 \times CH$), 133.9 (C), 141.89 (C), 185.3 (CO).

Compound 7. Yield (0.143 g, 65%); IR: 2150 (SCN), 1689 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.62$ (s, 2H), 7.81 (d, 2H), 8.38 (d, 2H), 12.98 (brs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 41.0$ (CH_2), 111.5 (SCN), 132.1 ($2 \times CH$), 134.0 ($2 \times CH$), 136.0 (C), 136.9 (C), 168.3 (COOH), 185.3 (CO). Anal. calcd. for $C_{10}H_7NO_3S$: C, 54.29; H, 3.19; N, 6.33; S, 14.49. Found: C, 54.37; H, 3.21; N, 6.39; S, 14.42.

Compound 8. Yield (0.136 g, 70%); IR: 2150 (SCN), 1687(CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.31$ (s, 2H), 7.35 (d, 2H), 7.89 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 41.0$ (CH_2), 111.5 (SCN), 115.3 ($2 \times CH$), 131.3 (C), 135.0 ($2 \times CH$), 160.8 (C), 164.7 (C), 185.3 (CO). Anal. calcd. for C_9H_6NOSF: C, 55.37; H, 3.09; N, 7.17; S, 16.43; F, 9.72. Found: C, 55.39; H, 4.01; N, 7.18; S, 16.48; F, 9.72.

Representative Examples of Spectra Data of Phenacyl Azide

Compound 9. Yield (0.181 g, 95%); IR: 2122 (N_3), 1687 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 3.81$ (s, 3H), 4.53 (s, 2H), 6.86 (d, 2H), 7.73 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 54.3$ (CH_3), 55.1 (CH_2), 116.2 ($2 \times CH$), 128.8 ($2 \times CH$), 132.7 (C), 162.7 (C), 193.1 (CO).

Compound 10. Yield (0.136 g, 85%); IR: 2104 (N_3), 1692 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.51$ (s, 2H), 7.36–7.56 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.1$ (CH_2), 126.2 ($2 \times CH$), 129.4 ($2 \times CH$), 131.1 (CH), 137.1 (C), 193.1 (CO).

Compound 11. Yield (0.144 g, 70%); IR: 2101 (N_3), 1697 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.53$ (s, 2H), 8.09 (d, 2H), 8.43 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.1$ (CH_2), 125.8 ($2 \times CH$), 128.1 ($2 \times CH$), 141.9 (C), 149.7 (C), 193.0 (CO).

Compound 12. Yield (0.220 g, 92%); IR: 2101 (N_3), 1699 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.52$ (s, 2H), 7.49 (d, 2H), 7.73 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.1$ (CH_2), 127.8 (C), 128.9 ($2 \times CH$), 133.9 ($2 \times CH$), 134.8 (C), 193.3 (CO). Anal. calcd. for $C_8H_6N_3OBr$: C, 39.95; H, 2.52; N, 17.47; Br, 33.29. Found: C, 39.99; H, 2.50; N, 17.49; Br, 33.49.

Compound 13. Yield (0.156 g, 80%); IR: 2102 (N_3), 1692 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.50$ (s, 2H), 7.44 (d, 2H), 7.66 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.1$ (CH_2), 128.1 (2 × CH), 129.9 (2 × CH), 136.9 (C), 139.03 (C), 193.2(CO).

Compound 15. Yield (0.149 g, 73%); IR: 2101 (N_3), 1699 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.53$ (s, 2H), 7.69 (d, 2H), 8.13 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.1$ (CH_2), 132.8 (2 × CH), 133.4 (2 × CH), 135.9 (C), 138.73 (C), 167.0 (COOH), 193.1 (CO). Anal. calcd. for $C_9H_7N_3O_3$: C, 52.68; H, 3.41; N, 20.49. Found: C, 52.57; H, 3.48; N, 20.53.

Compound 16. Yield (0.125 g, 70%); IR: 2102 (N_3), 1695 (CO) (cm^{-1}); ^1H NMR (400 MHz, $CDCl_3$): $\delta = 4.53$ (s, 2H), 7.29 (d, 2H), 7.83 (d, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): $\delta = 55.30$ (CH_2), 117.8 (2 × CH), 131.56 (2 × CH), 133.45 (C), 160.7 (C), 165.8 (C), 193.1 (CO). Anal. calcd. for $C_8H_6N_3oF$: C, 53.63; H, 3.37; N, 23.45; F, 10.61. Found: C, 53.57; H, 3.34; N, 23.48; F, 10.7.

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