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Preparation of 3,3-bis(ethylthiol)-2-arylacrylonitrile and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile via an electrogenerated base-promoted reaction

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Preparation of 3,3-bis(ethylthiol)-2-arylacrylonitrile and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile via an electrogenerated base-promoted reaction

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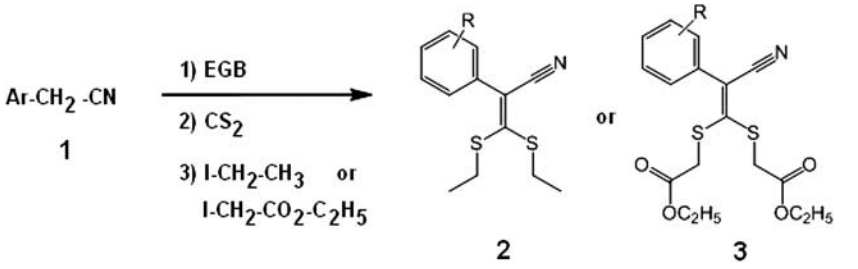
A simple method for the preparation of 3,3-bis(ethylthiol)-2-arylacrylonitrile and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile is described. The electrogenerated cyanomethyl base/anion obtained from electroreduction of acetonitrile promotes reactions between an arylacetonitrile, carbon disulfide and ethyl iodide or ethyl 2-chloroacetate. The products are obtained in good yields under mild reaction conditions with respect to previously reported methods. The effect of the number of Faradays and mechanism of the reaction are discussed.



Keywords: 3,3-bis(ethylthiol)-2-arylacrylonitrile; 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile; carbon disulfide; arylacetonitrile; electrogenerated base

Introduction

Thanks to their prominence in the synthesis of several biologically active molecules, such as fungicidals, flavonoid pigments, and sexual pheromones, numerous studies have been devoted to the exploration of the reactivity of arylacetonitriles (*1–5*). However, in order to avoid the use of polluting solvents and to circumvent the difficulties of the experimental protocols involved in

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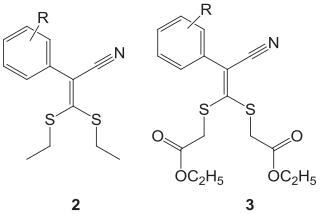
classic syntheses, new approaches under very mild conditions are needed. In the literature, the condensations of arylacetonitriles with carbonyls, isothiocyanates, and 1,2 – and 1,3 – dihalogenated alkanes are based on the traditional Knovenagel reaction which use strongly basic alkalai hydroxides or hydrides (*6*). Recently, it was found these bases could be substituted by electrogenerated bases (EGBs) to promote reactions sometimes in higher yields and under milder conditions (*7–9*). Electrogenerated species-promoted reactions in general proceed smoothly with an easy work-up and do not require harsh reaction conditions such as high temperatures or the use of highly reactive reagents. EGBs were initially mentioned by Baizer and coworkers (*10*) and recently used by Feroci *et al*. (*11–16*) for the preparation of carbonates, carbamates and oxazolines in two compartments cell.

In the past few years, we have focused on the development of an alternative synthesis of acyclic dithiocarbamates, N-benzylic rhodanines, and thiazolidine-2-thiones, respectively, from primary and secondary aliphatic and benzylic amines, carbons disulfide, alkyl halides, 2-bromoacetate and 2-chloroacetone using *in situ* EGBs (*7–9*). To the best of our knowledge, no electrochemical method has been published to date on the reaction between arylacetonitrile and electrogenerated species. Hence, the aim of this study is to develop the reactivity of these compounds by using EGBs. The study reports the electrochemical promoted synthesis of 3,3-bis(ethylthiol)-2-arylacrylonitrile (**2**) and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile (**3**) starting from arylacetonitrile (Scheme 1) A series of compounds were prepared and the effect of the number of Faradays and the mechanism are discussed.



2 3

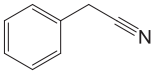
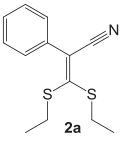
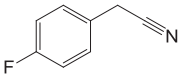
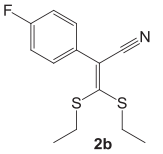
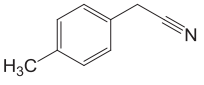
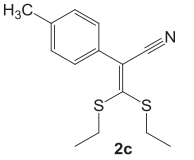
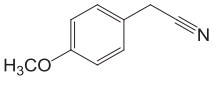
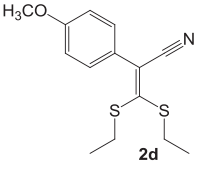
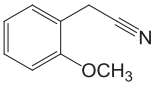
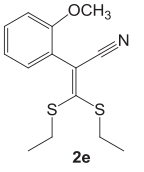
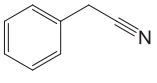
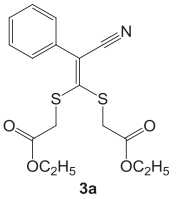
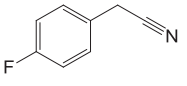
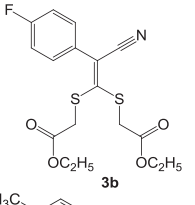
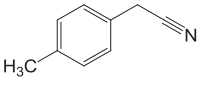
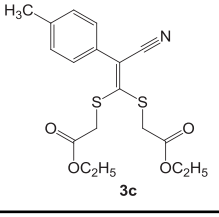
Scheme 1. Synthesized products (**2**) and (**3**) from arylacetonitrile via EGB-assisted reaction.

Results and discussion

Cyclic voltammetry of arylacetonitrile (**1**) in tetrabutylammonium tetrafluoroborate acetonitrile solution (0.1 mol/L) on a platinum electrode show that arylacetonitriles are not appreciably electroactive and are not reduced which means that they can be introduced either in the beginning (method A) or at the end of the electrolysis (method B) according to Feroci *et al*. (*17*) procedure.

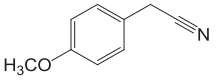
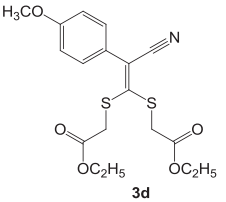
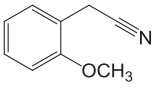
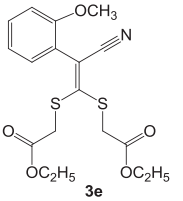
Electrolysis at constant current density in the presence of tetrabutylammonium tetrafluoroborate (0.1 mol/L) as the supporting electrolyte, in an undivided cell cooled at $-20\,^{\circ}C$ fitted with a magnesium rod as the anode and a stainless steel grid cathode, under an inert atmosphere of nitrogen, probably yields the arylacetonitrile with the cyanomethyl carbanions. The electrolysis is stopped approximately after 3–4 h of reaction. This time is enough to permit the formation of 5×10^{-3} mole of EGB. Then, carbon disulfide is added immediately to the solution followed by ethyl iodide or ethyl-2-chloroacetate 15 min later. The mixture is continually stirred over night at ambient temperature. The products are isolated following standard work-up procedure (*6–8*) and purified by column chromatography. All the resulting products reported in Table 1 were identified by examination of their spectroscopic data.

Table 1. Synthesized 3,3-bis(ethylthiol)-2-arylacrylonitrile (**2**) and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile (**3**).

Entry	Starting arylacetonitrile	Electrophile	Product	Q^a	Yield[b] (%)
1		A	 **2a**	2	71
2		A	 **2b**	2	75
3		A	 **2c**	2	76
4		A	 **2d**	2	69
5		A	 **2e**	2	65
6		B	 **3a**	2	62
7		B	 **3b**	2	60
8		B	 **3c**	2	59

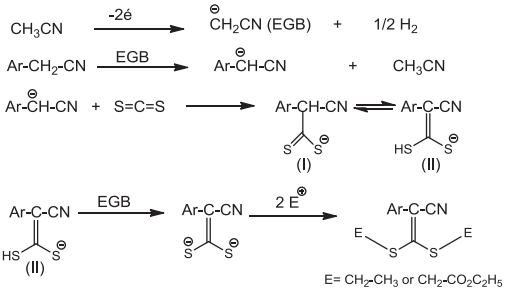
(*Continued*).

Table 1. Continued.

Entry	Starting arylacetonitrile	Electrophile	Product	Q^a	Yield[b] (%)
9		B	 **3d**	2	68
10		B	 **3e**	2	73

Notes: A, ethyliodide; B, ethyl 2-chloroacetate. [a]The consumed quantity of electricity represents the number of Faradays per mole of arylacetonitrile. [b]The yield was calculated in regard to the starting arylacetonitrile.

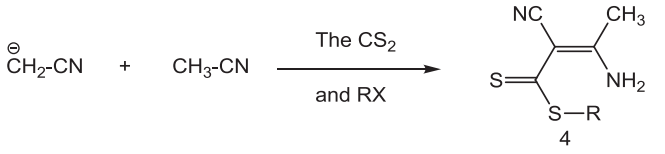
The EGB is presumed to be, in the present case, primarily the cyanomethyl carbanion, formed along with dihydrogen by the reduction of acetonitrile (*7–9*) at the stainless steel grid cathode and stabilized with the electrogenerated Mg^{2+} cations formed by oxidation of the sacrificial magnesium anode. The EGB deprotonates the arylacetonitrile to yield a second anion that reacts with CS_2 to yield the corresponding Mg dithiocarbonic salt (I).



Scheme 2. Proposed mechanism of the preparation of products (**2**) and (**3**) by EGB-promoted reaction.

The intermediate anion (I) probably tautomerizes to give anion (II) that is preferentially stabilized by conjugation. A second EGB removes the mobile proton of the –SH group to yield the dianions. Finally, alkylation by two equivalents of ethyl iodide or ethyl 2-chloroacetate yields the products (**2**) and (**3**), respectively.

Phenylacetonitrile is chosen as the standard starting reagent in order to study the effect of the number of Faradays on the reaction yield (Table 2). It was found that the yield increases as the number of Faradays increases to reach a satisfactory yield of 76% at ca. 2 Faraday. When the number of Faraday is ca. 5 the yield is improved only by 10%. So, 2.0–2.5 Faraday seems a good compromise between a good yield of the reaction and the current consumption. Moreover, at 5 Faraday a byproduct (**4**) yield, formed by the autocondensation reaction of acetonitrile, increases dramatically (Scheme 3) (*8*).



Scheme 3. Preparation of byproduct **(4)** by reaction of autocondensation of acetonitrile.

Table 2. Variation of the yield of **2c** as a function of the consumed number of Faradays in respect to the amount of arylacetonitrile.

Entry	Q/F (mol^{-1})	Yield of **2c** (%)	Time (min)
1	1	56	100
2	2	76	200
3	3	82	300
4	4	85	400
5	5	86	500

The amount of acetonitrile is reduced by evaporation and the resulting mixture was extracted three times with 50 ml ether. The ethereal phase was washed by small amounts of water and dried over magnesium sulfate. The ether was removed and the residue was purified by column chromatography on silica gel 60 using ethyl acetate/petrol ether (v:v = 1:9) as eluent. Products (**2**) and (**3**) were identified by IR, NMR spectroscopy, GC–MS and elemental analysis.

Conclusion

In conclusion, the use of an EGB provides a new electrochemical methodology that allows the preparation of 3,3-bis(ethylthiol)-2-arylacrylonitriles and 3,3-bis(ethoxyacetatethiol)-2-arylacrylonitrile (**3**) from arylacetonitrile, carbon disulfide and ethyliodide or ethyl-2-chloroacetate in high yields. The reaction occurs under mild conditions and avoids the use of polluting or hazardous chemicals or the addition of a base. The mechanism of the reaction is investigated by cyclic voltammetry and the effects of number of Faradays on the reaction yield have been studied.

Experimental

3,3-bis(ethylthio)-2-phenylacrylonitrile (2a)

Yield: 71%: IR (cm^{-1}, $CHCl_3$)ν: 2220 (CN); 1600 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.10 (t, 3H, CH_3); 1.25 (t, 3H, CH_3); 2.60 (q, 2H, CH_2); 2.90 (q, 2H, CH_2); 7.20–7.40 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 13.65; 13.76; 28.31; 29.06; 114.38; 117.41; 126.04; 126.33; 126.46; 126.87; 127.26; 127.80; 133.19; 153.50. MS (EI) m/z: 77 (10); 89 (16); 105 (30); 115 (15); 133 (20); 159 (100); 160 (45); 192 (25); 220 (12); 249 (80). Elemental analysis: %C =/60.01; %H = 5.82; %N = 5.40 (experimental). %C = 62.61; %H = 6.06; %N = 5.62 (calculated).

3,3-bis(ethylthio)-2-(4-fluorophenyl)acrylonitrile (2b)

Yield: 75%: IR (cm^{-1}, $CHCl_3$)ν: 2226 (CN); 1608 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.20 (t, 3H, CH_3); 1.30 (t, 3H, CH_3); 2.75 (q, 2H, CH_2); 2.95 (q, 2H, CH_2); 6.90–7.40 (mu, 4H, CH

arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.69; 14.76; 29.82; 30.14; 115.51; 117.22; 128.42; 129.31; 129.55; 129.72; 130.25; 131.28; 154.88; 160.89. ^{19}F NMR (282.37 MHz, $CDCl_3$) δ: −110.23 (s, 1F, CF arom.). MS (EI) m/z: 61 (10); 105 (24); 133 (32); 151 (20); 177 (100); 178 (40), 210 (30); 238 (11); 267.1 (80). Elemental analysis: %C=/60.53; %H = 5.46; %N = 5.62 (experimental). %C = 58.39; %H = 5.28; %N = 5.24 (calculated).

3,3-bis(ethylthio)-2-(p-tolyl)acrylonitrile (2c)

Yield: 76%: IR (cm^{-1}, $CHCl_3$) ν: 2222 (CN); 1608 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.20 (t, 3H, CH_3); 1.40 (t, 3H, CH_3); 2.40 (s, 3H, CH_3); 2.75 (q, 2H, CH_2); 3.10 (q, 2H, CH_2); 7.20–7.40 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.75; 14.67; 21.33; 29.25; 30.07; 115.80; 118.50; 128.55; 128.66; 129.08; 129.21; 129.67; 129.72; 138.92; 153.44. MS (EI) m/z: 91.1 (20); 119.1 (18); 148.0 (14.5); 172.0 (38); 173.0 (100); 174.0 (34.5); 206.0 (16); 234.0 (11); 263.1 (80). Elemental analysis: %C=/59.93; %H = 6.34; %N = 5.88 (experimental). %C = 63.83; %H = 6.50; %N = 5.32 (calculated).

3,3-bis(ethylthio)-2-(4-methoxyphenyl)acrylonitrile (2d)

Yield: 69%: IR (cm^{-1}, $CHCl_3$) ν: 2221 (CN); 1606 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.15 (t, 3H, CH_3); 1.30 (t, 3H, CH_3); 2.70 (q, 2H, CH_2); 2.90 (q, 2H, CH_2); 3.70 (s, 3H, CH_3); 6.80–7.35 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.71; 14.72; 29.32; 29.66; 55.22; 113.42; 113.91; 114.54; 126.56; 126.60; 129.05; 129.77; 152.47; 159.85. MS (EI) m/z: 69 (10); 102 (11); 146 (24); 167 (30); 189.0 (100); 199 (20) 250 (10); 279.1 (49). Elemental analysis: %C=/61.33; %H = 5.98; %N = 5.26 (experimental). %C = 60.18; %H = 6.13; %N = 5.01 (calculated).

3,3-bis(ethylthio)-2-(2-methoxyphenyl)acrylonitrile (2e)

Yield: 65%: IR (cm^{-1}, $CHCl_3$) ν: 2227 (CN); 1602 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.25 (t, 3H, CH_3); 1.45 (t, 3H, CH_3); 2.70 (q, 2H, CH_2); 3.00 (q, 2H, CH_2); 3.8 (s, 3H, CH_3); 7.20 –7.40 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.75; 14.77; 28.88; 29.71; 55.54; 110.54; 111.76; 117.50; 126.50; 126.60; 129.06; 129.17; 155.54; 156.78. MS (EI) m/z: 146 (25); 174 (28); 189.0 (100); 250 (10); 279 (50). Elemental analysis: %C=/60.72; %H = 6.04; %N = 5.39 (experimental). %C = 60.18; %H = 6.13; %N = 5.01 (calculated).

3,3-bis(ethoxyacetatethiol)-2-phenylacrylonitrile (3a)

Yield: 62%: IR (cm^{-1}, $CHCl_3$) ν: 2225 (CN); 1730 (C=O); 1604 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.10 (t, 3H, CH_3); 1.20 (t, 3H, CH_3); 3.50 (s, 2H, CH_2); 3.70 (s, 2H, CH_2); 4.10 (q, 2H, CH_2); 4.20 (q, 2H, CH_2); 7.30–7.40 (mu, 5H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.00; 14.05; 36.31; 36.60; 61.97; 62.04; 117.53; 119.32; 128.12; 128.29; 128.71; 128.91; 129.06; 133.51; 151.32; 168.09; 168.25. MS (EI) m/z: 77 (13); 115 (27); 159 (100); 160 (50); 204 (100); 205 (40); 232 (19); 278 (43); 365.1 (48). Elemental analysis: %C=/56.15; %H = 5.37; %N = 3.76 (experimental). %C = 55.87; %H = 5.24; %N = 3.83 (calculated).

3,3-bis(ethoxyacetatethiol)-2-(4-fluorophenyl)acrylonitrile (3b)

Yield: 60%: IR (cm^{-1}, $CHCl_3$) ν: 2225 (CN); 1731 (C=O); 1605 (C=C). ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.30 (t, 3H, CH_3); 1.40 (t, 3H, CH_3); 3.60 (s, 2H, CH_2); 3.80 (s, 2H, CH_2); 4.20 (q,

2H, CH_2); 4.30 (q, 2H, CH_2); 7.10–7.60 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.08; 14.11; 36.31; 36.80; 62.10; 62.13; 115.77; 116.06; 117.47; 118.46; 129.52; 129.56; 131.24; 131.35; 151.61; 168.09; 168.20. ^{19}F NMR (282.37 MHz, $CDCl_3$) δ: −110.24 (s, 1F, CF arom.). MS (EI) m/z: 61 (10); 105 (24); 133 (32); 151 (20); 177 (100); 178 (40), 222 (100); 223 (51) 238 (11); 383.1 (50). Elemental analysis: %C=/54.53; %H = 5.07; %N = 3.87 (experimental). %C = 53.25; %H = 4.73; %N = 3.65 (calculated).

3,3-bis(ethoxyacetatethiol)-2-(p-tolyl)acrylonitrile (3c)

Yield: 59%: IR (cm^{-1}, $CHCl_3$)ν: 2223 (CN); 1735 (C=O); 1606 (C=C). ^1H NMR (300 MHz, $CDCl_3$) δ: 1.30 (t, 3H, CH_3); 1.40 (t, 3H, CH_3); 2.40 (s, 3H, CH_3); 3.60 (s, 2H, CH_2); 3.80 (s, 2H, CH_2); 4.20 (q, 2H, CH_2); 4.30 (q, 2H, CH_2); 7.20–7.40 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.60; 14.69; 23.22; 36.25; 36.87; 62.50; 62.53; 114.65; 118.65; 127.36; 128.36; 129.51; 129.60; 131.52; 135.63; 151.44; 167.96; 168.09. MS (EI) m/z: 91.1 (25); 119.1 (11); 172.0 (45); 173.0 (100); 174.0 (36); 206.0 (16); 218 (100); 219 (49); 379.1 (40). Elemental analysis: %C=/56.03; %H = 5.16; %N = 3.40 (experimental). %C = 56.97; %H = 5.58; %N = 3.69 (calculated).

3,3-bis(ethoxyacetatethiol)-2-(4-methoxyphenyl)acrylonitrile (3d)

Yield: 68%: IR (cm^{-1}, $CHCl_3$)ν: 2226 (CN); 1736 (C=O); 1606 (C=C). ^1H NMR (300 MHz, $CDCl_3$) δ: 1.20 (t, 3H, CH_3); 1.30 (t, 3H, CH_3); 3.60 (s, 2H, CH_2); 3.80 (s, 2H, CH_2); 3.90 (s, 3H, CH_3); 4.20 (q, 2H, CH_2); 4.30 (q, 2H, CH_2); 7.00–7.50 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.07; 14.10; 36.32; 36.80; 55.37; 61.98; 62.03; 114.07; 115.03; 117.75; 119.54; 125.75; 128.82; 130.65; 149.00; 160.34; 168.19; 168.33. MS (EI) m/z: 102 (15); 146 (29); 167 (30); 189.0 (100); 199 (20); 234 (100); 235 (56); 395.1 (45). Elemental analysis: %C=/55.10; %H = 5.67; %N = 3.82 (experimental). %C = 54.66; %H = 5.35; %N = 3.54 (calculated).

3,3-bis(ethoxyacetatethiol)-2-(2-methoxyphenyl)acrylonitrile (3e)

Yield: 73%: IR (cm^{-1}, $CHCl_3$)ν: 2224 (CN); 1735 (C=O); 1603 (C=C). ^1H NMR (300 MHz, $CDCl_3$) δ: 1.20 (t, 3H, CH_3); 1.30 (t, 3H, CH_3); 3.60 (s, 2H, CH_2); 3.80 (s, 2H, CH_2); 3.90 (s, 3H, CH_3); 4.20 (q, 2H, CH_2); 4.30 (q, 2H, CH_2); 7.00–7.20 (mu, 4H, CH arom.). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.10; 14.12; 36.45; 36.91; 54.97; 62.08; 62.15; 114.25; 115.17; 118.05; 119.61; 125.60; 128.92; 131.05; 148.89; 159.43; 168.21; 168.30. MS (EI) m/z: 102 (18); 146 (30); 167 (41); 189.0 (100); 199 (20); 234 (100); 235 (49); 395.1 (50). Elemental analysis: %C=/54.22; %H = 5.62; %N = 3.73 (experimental). %C = 54.66; %H = 5.35; %N = 3.54 (calculated).

3-amino-2-cyanobut-2-enedithioate ethyl (4a)

IR (cm^{-1}, $CHCl_3$)ν: 3330 (NH_2); 2189 (CN); 1600 (C=C); 1192 (C=S). ^1H NMR (300 MHz, $CDCl_3$) δ: 1.30 (t, 3H, CH_3); 2.40 (s, 3H, CH_3); 3.2 (q, 2H, CH_2); 6.5 (s, 2H, NH_2). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 12.02; 24.55; 29.01; 93.66; 118.25; 167.17; 212.02.

2-((3-amino-2-cyanobut-2-enethioyl)thio) acetate ethyl (4b)

IR (cm^{-1}, $CHCl_3$)ν: 3334 (NH_2); 2193 (CN); 1734 (C=O); 1606 (C=C); 1201 (C=S). ^1H NMR (300 MHz, $CDCl_3$) δ: 1.29 (t, 3H, CH_3); 2.20 (s, 3H, CH_3); 3.80 (s, H, CH); 4.10 (q, 2H, CH_2)

8.60 (s, 2H, NH_2). ^{13}C NMR (75.47 MHz, $CDCl_3$) δ: 14.10; 23.34; 39.50; 61.01; 69.22; 115.06; 168.62; 174.66; 220.02.

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