# Synthesis of corrosion preventive long-chain *N*-alkyl-2-(phenylthio)acetohydrazides and 2-oxo-2-phenylethyl-2-alkanoylhydrazinecarbodithioates

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Abstract Long-chain *N*-alkyl-2-(phenylthio)acetohydrazides were synthesized *via* the reactions of 2-(phenylthio)acetohydrazide with long-straight-chain aldehydes and then reduction with sodium borohydride. The reactions of long-straight-chain hydrazides with carbon disulfide in alkaline media give the corresponding carbodithioate salts. Heating of potassium 2-alkanoylhydrazinecarbodithioates with phenacyl bromide do not yield cyclization and failed to give the corresponding long-chain thiazolidine-2-thiones, but gave the corresponding 2-oxo-2-phenylethyl-2-alkanoylhydrazinecarbodithioates *via* nucleophilic substitution reaction. In addition, the synthesized compounds were tested for their corrosion prevention capabilities in acidic or in mineral oil media.

**Keywords** Fatty acids; Surface; Corrosion inhibitors; Acidic; Mineral oil media.

## Introduction

Long-chain fatty acids and compounds derived from them, especially, N, S, or P derivatives find wide applications in every area of industrial activity while many of them are products or constituents of products. Fat-based emulsifiers, fabric softeners and conditioners, synthetic lubricants, oil field chemicals, derivatives using in metalworking fluids, flotation, textiles, cosmetics, and toiletries are examples [1].

The largest use of N and/or S containing fatty acids derivatives in oil field chemicals is in corrosion inhibitors. The prior art has disclosed a wide variety of chemical compounds which effectively reduce the corrosion. Fatty diethanolamides [2], long-chain imidazolines [3], isoxazolines and isoxazolidines [4], mercapto oxadiazoles and triazoles [5, 6], and thiosemicarbazides [7] are well known compounds in corrosion literature. In general, S-containing derivatives are among the most effective inhibitors. This is due to the fact that lone electron pairs at sulfur can easily be polarized.

We report herein the synthesis of fatty *N*-alkyl-2-(phenylthio)acetohydrazides 4a-4c and 2-oxo-2-phenylethyl-2-alkanoylhydrazinecarbodithioates 7a-7d. It was found that under the reaction conditions tested carbodithioates 6a-6d give the 2-oxo-2-phenylethyl-2 alkanoylhydrazine carbodithioates 7a-7d and do not yield cyclization to afford thiazolidine-2-thiones 8a-8d. We also investigate the corrosion prevention properties of the synthesized compounds in 2M HCl acidic media or paraffin based mineral oil media.

# **Results and discussion**

There are many papers related to the reactions between a variety of substrates and the products

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obtained from carboxylic acid hydrazides and carbon disulfide in alcoholic alkaline media [8–11]. The starting material 1 was synthesized by first reacting thiophenol with sodium chloroacetate in alcoholic alkaline media to give the sodium salt of 2-(phenylthio)acetic acid, which provided the desired acid after acidification [12]. Then this acid was refluxed with methanol in the presence of a little amount of sulfuric acid to give the methyl ester. Treatment of this ester with hydrazine hydrate in boiling methanol gave the corresponding hydrazide 1 in good yield. The IR spectrum shows the characteristic bands for  $-NHNH_2$  at  $\bar{\nu} = 3290 \text{ cm}^{-1}$ and for -NC=O at  $\bar{\nu} = 1649 \text{ cm}^{-1}$ . Next, the treatment of 1 with an equivalent of 2a-2c in methanol yielded the corresponding N-alkylidene-2-(phenylthio)acetohydrazides 3a-3c in moderate to good yields (Scheme 1).

The IR spectra of **3a–3c** showed absorption bands at  $\bar{\nu} = 3222 \text{ cm}^{-1}$  due to the –NH group, and at  $\bar{\nu} = 3080 \text{ cm}^{-1}$  due to the Ar=C–H stretching, at  $\bar{\nu} = 1665 \text{ cm}^{-1}$  due to the -N=C group. The <sup>1</sup>H NMR spectrum showed a singlet at  $\delta = 9.42 \text{ ppm}$  for the NH proton, a triplet at  $\delta = 7.11 \text{ ppm}$  for the -N=CH- proton and singlet at  $\delta = 4.01 \text{ ppm}$  for the *Ph*SCH<sub>2</sub> protons. Compounds **3a–3c** were reduced with sodium borohydride in methanol to give the corresponding hydrazides **4a–4c** (Scheme 1). The compound **4a–4c** showed characteristic IR bands at  $\bar{\nu} = 3287 \text{ cm}^{-1}$  and at  $\bar{\nu} = 3215 \text{ cm}^{-1}$  for the  $-NHNH_2$  group, at  $\bar{\nu} = 1650 \text{ cm}^{-1}$  for the  $-CONHNH_2$  carbonyl group. The <sup>1</sup>H NMR spectrum showed a singlet at  $\delta = 3.65 \text{ ppm}$  for the  $-SCH_2-$  protons and a triplet at  $\delta = 2.71 \text{ ppm}$  for the  $-NNCH_2-$  protons.

The starting long-straight-chain carboxylic acid hydrazides 5a-5d were simply prepared from the corresponding methyl esters and hydrazine hydrate in boiling methanol with good yields [13]. Treatment of the obtained hydrazides 5a-5d with carbon disulfide and potassium hydroxide in ethanol gave the salts of carbodithioates 6a-6d, which were used in the next step without further purification (Scheme 2).



Scheme 2

The IR spectrum of **6a–6d** showed absorption bands at  $\bar{\nu} = 3448 \,\mathrm{cm}^{-1}$  (br band) for the –NH groups and at  $\bar{\nu} = 1647 \,\mathrm{cm}^{-1}$  for the C=O group. Decomposition of these salts was observed upon heating to high temperatures. Next, carbodithioates 6a-6d and phenacyl bromide were heated in ethanol. They failed to give the desired thiazolidine-2-thiones 8a-8d, but the corresponding 2-oxo-2-phenylethyl-2-alkanovlhydrazinecarbodithioates 7a-7d were obtained (Scheme 2). The IR spectrum of compounds 7a-7d showed absorption bands at  $\bar{\nu} = 3444 \,\mathrm{cm}^{-1}$  (br band) for the -NH groups, and at  $\bar{\nu} = 1693$  and  $1682 \text{ cm}^{-1}$  for the C=O groups. The <sup>1</sup>H NMR spectrum does not exhibit a singlet signal for 8 thiazolidine H-5 proton, but showed a singlet at  $\delta =$ 4.91 ppm for two of the -SCSCH<sub>2</sub>- protons related to 7a-7d. This result proves that the expected cyclization shown in Scheme 2 not observed.

#### Corrosion inhibition

In order to investigate corrosion preventive properties of the synthesized 4a-4c and 7a-7d, corrosion tests were performed using 2M HCl and mineral oil media. All compounds exhibited corrosion preventive capability (Table 1).

Inhibitor molecules bearing S atoms in their structures display improved efficiencies [4]. Inhibitor molecules are adsorbed at the metal surface. A pos-

**Table 1** Corrosion inhibition efficiencies for 4a-4c (inhibitor concentration  $\delta = 100 \text{ ppm}$ ) in 2*M* HCl for 25 h at 30°C\*

Inhibition efficiencies
95.4
97.0
97.5

\* Duplicate experiments were performed in each case and the mean value of the weight loss is reported

sible orientation of the acetohydrazides 4a-4c at the metal-acidic solution interface is shown in Fig. 1.

For the corrosion inhibitors 7a-7d no visible rust spots were observed on the metal stripe and it can be concluded that the tested organic compounds may be suitable inhibitors for such a corrosive medium.

## **Experimental**

Melting points were recorded by Büchi melting point B-540 apparatus. IR spectra were measured by Nicolet FTIR 6700 spectrometer. <sup>1</sup>H NMR spectra were measured using a Varian mercury plus spectrometer (400 MHz) in CDCl<sub>3</sub> using *TMS* as an internal standard. All reagents and solvents were purchased from either Merck or Fluka Chemie and used without further purification. 2-(Phenylthio)acetic acid was synthesized according to Ref. [12], and long-straight-chain carboxylic acid hydrazides **5a–5d** were prepared as described in Ref. [13]. Phenacyl bromide was prepared by a literature method [14].

#### 2-(Phenylthio)acetohydrazide (1, C<sub>8</sub>H<sub>10</sub>N<sub>2</sub>OS)

To a solution of 20 g 2-(phenylthio)acetic acid (118.9 mmol) in 300 cm<sup>3</sup> methanol, 7 cm<sup>3</sup> sulfuric acid were added cautiously. The observed clear solution was refluxed for 3-3.5 h at 65°C. Solvent was removed by using a rotary evaporator and ice water was added into the flask. Crude ester was extracted with  $3 \times 50 \text{ cm}^3$  ether. The layers were separated and the organic layer was washed successively with 10% NaHCO<sub>3</sub>, water, and dried over Na<sub>2</sub>SO<sub>4</sub>. Ether was removed by using a rotary evaporator to give 19.4 g colorless oily liquid methyl 2-(phenylthio)acetate which was dissolved in  $50 \,\mathrm{cm}^3$ methanol, 5.33 g hydrazine hydrate (106.5 mmol) were added, and the resulting mixture was refluxed for 3 h at 60-65°C. Methanol was removed by using a rotary evaporator. The obtained white solid was crystallized from ethyl acetate/ petroleum ether and 14.6g white needles of 1 (67% for 2 steps) were collected. Mp 63–64°C; IR (KBr):  $\bar{\nu} = 3290$  $(NHNH_2)$ , 1649 (NC=O) cm<sup>-1</sup>.

### *N-Alkylidene-2-(phenylthio)acetohydrazides* **3a–3c**, representative procedure for **3c**

To a stirred solution of 1 g 2-(phenylthio)acetohydrazide (1) (5.49 mmol) in  $15 \text{ cm}^3$  methanol, 1.01 g of dodecanal (2c) (5.48 mmol) were added and the resulting mixture was stirred



Fig. 1 Possible orientations of the inhibitor molecules 4c at the metal-acidic solution interface

at room temperature for 30 min. Then it was heated at  $60^{\circ}\text{C}$  for 2 h. Methanol was evaporated using a rotary evaporator to give 1.60 g of a white solid. If desired it can be recrystallized from methanol to give **3c** as a white crystalline solid.

### N-Octylidene-2-(phenylthio)acetohydrazide

 $(3a, C_{16}H_{24}N_2OS)$ 

Yield 69%; mp 54–55°C; IR (KBr):  $\bar{\nu} = 3211$  (NH), 3067 (Ar, =CH), 1665 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 9.41$ (s, CONH), 7.46–7.43 (m, 1H, Ar), 7.37–7.17 (m, 4H, Ar), 7.10 (t, J = 5.2 Hz, -N=CH), 4.01 (s, *Ph*SCH<sub>2</sub>), 2.34 (m, -N=CHC<u>H<sub>2</sub></u>), 1.50 (quin, J = 7.2 Hz, -N=CHCH<sub>2</sub>C<u>H<sub>2</sub></u>), 1.31–1.26 (m, 8H, -CH<sub>2</sub>–), 0.89 (t, J = 7.2 Hz, -CH<sub>3</sub>) ppm.

#### N-Decylidene-2-(phenylthio)acetohydrazide

 $(3b, C_{18}H_{28}N_2OS)$ 

Yield 97%; mp 70–71°C; IR (KBr):  $\bar{\nu} = 3213$  (NH), 3067 (Ar, =CH), 1677 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 9.40$  (s, CONH), 7.46–7.44 (m, 1H, Ar), 7.37–7.17 (m, 4H, Ar), 7.10 (t, J = 5.2 Hz, -N=CH), 4.01 (s, PhSCH<sub>2</sub>), 2.34 (m, -N=CHCH<sub>2</sub>), 1.50 (quin, J = 6.8 Hz, -N=CHCH<sub>2</sub>CH<sub>2</sub>), 1.30–1.25 (m, 12H, -CH<sub>2</sub>–), 0.88 (t, J = 7.2 Hz, -CH<sub>3</sub>) ppm.

#### *N*-Dodecylidene-2-(phenylthio)acetohydrazide (**3c**, C<sub>20</sub>H<sub>32</sub>N<sub>2</sub>OS)

Yield 75% (1.44 g); mp 72–73°C; IR (KBr):  $\bar{\nu} = 3222$  (NH), 3080 (Ar, =CH), 1665 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 9.42$  (s, CONH), 7.46–7.43 (m, 1H, Ar), 7.36–7.17 (m, 4H, Ar), 7.11 (t, J = 5.2 Hz, -N=CH), 4.01 (s, *Ph*SCH<sub>2</sub>), 2.34 (m, -N=CHC<u>H<sub>2</sub></u>), 1.50 (quin, J = 7.6 Hz, -N=CHCH<sub>2</sub>C<u>H<sub>2</sub></u>), 1.30–1.25 (m, 16H, -CH<sub>2</sub>–), 0.88 (t, J = 7.2 Hz, -CH<sub>3</sub>) ppm.

# *N-Alkyl-2-(phenylthio)acetohydrazides* **4***a***–4***c*; *representative procedure for* **4***c*

To a cooled and stirred solution of 1.44 g *N*-dodecylidene-2-(phenylthio)acetohydrazide **3c** (4.13 mmol) in tetrahydrofuran/methanol  $10/10 \text{ cm}^3$ , 0.25 g sodium borohydride (6.61 mmol) were added portionwise at  $5-10^{\circ}$ C during 10 min. It was stirred for 15 min at this temperature and for additional 1 h at room temperature. After **3c** was consumed (TLC control) the solvent was removed by using a rotary evaporator to give a white solid. This solid product was washed with cold water and dried overnight at room temperature. Recrystallization from methanol afforded the title compound **4c** as a white solid.

#### *N-Octyl-2-(phenylthio)acetohydrazide* (4a, C<sub>16</sub>H<sub>26</sub>N<sub>2</sub>OS)

Yield 63%; mp 78°C; IR (KBr):  $\bar{\nu} = 3286$  (NH), 3214 (NH), 3012 (Ar, =CH), 1650 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 7.99$  (s, -CONH), 7.31–7.29 (m, 4H, Ar), 7.24–7.20 (m, 1H, Ar), 4.55 (s, NNH), 3.65 (s, -SCH<sub>2</sub>), 2.71 (t, J = 7.6 Hz, -NNCH<sub>2</sub>), 1.36–1.23 (m, 12H, -CH<sub>2</sub>–), 0.88 (t, J = 6.8 Hz, -CH<sub>3</sub>) ppm.

*N-Decyl-2-(phenylthio)acetohydrazide* (**4b**,  $C_{18}H_{30}N_2OS$ ) Yield 63%; mp 78–78.7°C; IR (KBr):  $\bar{\nu} = 3286$  (NH), 3215 (NH), 3010 (Ar, =CH), 1649 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  = 7.99 (s, -CONH), 7.31–7.28 (m, 4H, Ar), 7.24–7.20 (m, 1H, Ar), 4.55 (s, NNH), 3.65 (s, -SCH<sub>2</sub>), 2.71 (t, J = 7.2 Hz, -NNCH<sub>2</sub>), 1.36–1.23 (m, 16H, -CH<sub>2</sub>–), 0.88 (t, J = 7.6 Hz, -CH<sub>3</sub>) ppm.

*N*-Dodecyl-2-(phenylthio)acetohydrazide (**4c**, C<sub>20</sub>H<sub>34</sub>N<sub>2</sub>OS) Yield 66% (0.95 g); mp 79–79.7°C; IR (KBr):  $\bar{\nu}$  = 3288 (NH), 3215 (NH), 3014 (Ar, =CH), 1651 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  = 7.98 (s, –CONH), 7.31–7.28 (m, 4H, Ar), 7.24– 7.20 (m, 1H, Ar), 4.54 (s, NNH), 3.65 (s, –SCH<sub>2</sub>), 2.71 (t, *J* = 7.2 Hz, –NNCH<sub>2</sub>), 1.36–1.23 (m, 20H, –CH<sub>2</sub>–), 0.88 (t, *J* = 7.2 Hz, –CH<sub>3</sub>) ppm.

# Potassium 2-alkanoylhydrazinecarbodithioates **6a–6d**; representative procedure for **6a**

To a solution of 0.8 g dodecanehydrazide **5a** (3.73 mmol) in  $20 \text{ cm}^3$  ethanol, 0.3 g potassium hydroxide (5.36 mmol) and 0.3 cm<sup>3</sup> carbon disulfide were added. The reaction mixture was stirred under reflux for 6 h and for additional one night at room temperature. Ethanol was evaporated by using a rotary evaporator to give a white powder.

#### Potassium 2-dodecanoylhydrazinecarbodithioate

#### $(6a, C_{13}H_{25}KN_2OS_2)$

Yield 74% (0.91 g); mp (dec); IR (KBr):  $\bar{\nu} = 3448$  (br, NH), 1647 (C=O) cm<sup>-1</sup>.

Yields of the **6b–6d** were 66, 70, and 61% with mp and IR data identical with those of **6a**.

### 2-Oxo-2-phenylethyl-2-alkanoylhydrazinecarbodithioates 7*a*-7*d*; representative procedure for 7*a*

To a solution of 0.9 g **6a** (2.74 mmol) in  $20 \text{ cm}^3$  ethanol, 0.55 g phenacyl bromide (2.76 mmol) were added and the resulting mixture was refluxed for 2 h. The mixture was cooled to room temperature and then ice cold water was added. The obtained yellow solid product was filtered under vacuum and left to dry at room temperature for one night. Solid material was crystallized from petroleum ether and washed with cold petroleum ether to give **7a** as a pale yellow crystalline compound.

# 2-Oxo-2-phenylethyl-2-dodecanoylhydrazinecarbodithioate (7a, $C_{21}H_{32}N_2O_2S_2$ )

Yield 68% (0.76 g); mp 59–60°C; IR (KBr):  $\bar{\nu} = 3444$  (br, NH), 3059 (Ar, =CH), 1693 (C=O), 1682 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.05-8.02$  (m, 2H, Ar), 7.66–7.62 (m, 1H, Ar), 7.53–7.50 (m, 2H, Ar), 4.91 (s, SCSCH<sub>2</sub>), 2.81 (t, J = 7.2 Hz, CH<sub>2</sub>CON), 1.76 (quin, J = 7.6 Hz,  $-CH_2-CH_2-CH_2-$ CH<sub>2</sub>–), 1.39–1.26 (m, 16H,  $-CH_2-$ ), 0.88 (t, J = 7.2 Hz,  $-CH_3$ ) ppm.

# $\label{eq:2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-tetradecanoylhydrazine-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-Oxo-2-phenylethyl-2-p$

carbodithioate (7b,  $C_{23}H_{36}N_2O_2S_2$ )

Yield 69%; mp 65–66°C; IR (KBr):  $\bar{\nu} = 3469$  (br, NH), 3058 (Ar, =CH), 1691 (C=O), 1685 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.05-8.03$  (m, 2H, Ar), 7.66–7.62 (m, 1H, Ar), 7.54–7.50 (m, 2H, Ar), 4.91 (s, SCSCH<sub>2</sub>), 2.82 (t, J = 7.6 Hz,

CH<sub>2</sub>CON), 1.76 (quin, J = 8.0 Hz,  $-CH_2-CH_2-CH_2-$ ), 1.39– 1.26 (m, 20H,  $-CH_2-$ ), 0.88 (t, J = 6.8 Hz,  $-CH_3$ ) ppm.

#### 2-Oxo-2-phenylethyl-2-hexadecanoylhydrazinecarbodithioate (7c, $C_{25}H_{40}N_2O_2S_2$ )

Yield 43%; mp 74–76°C; IR (KBr):  $\bar{\nu} = 3471$  (br, NH), 3058 (Ar, =CH), 1691 (C=O), 1682 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.05-8.03$  (m, 2H, Ar), 7.66–7.62 (m, 1H, Ar), 7.54–7.50 (m, 2H, Ar), 4.91 (s, SCSCH<sub>2</sub>), 2.82 (t, J = 7.6 Hz, CH<sub>2</sub>CON), 1.76 (quin, J = 7.2 Hz,  $-CH_2-CH_2-CH_2-$ ), 1.40–1.26 (m, 24H,  $-CH_2-$ ), 0.88 (t, J = 7.2 Hz,  $-CH_3$ ) ppm.

#### 2-Oxo-2-phenylethyl-2-octadecanoylhydrazinecarbodithioate (**7d**, C<sub>27</sub>H<sub>44</sub>N<sub>2</sub>O<sub>2</sub>S<sub>2</sub>)

Yield 34%; mp 77–78°C; IR (KBr):  $\bar{\nu} = 3469$  (br, NH), 3059 (Ar, =CH), 1691 (C=O), 1682 (C=O) cm<sup>-1</sup>; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta = 8.05-8.03$  (m, 2H, Ar), 7.66–7.62 (m, 1H, Ar), 7.54–7.50 (m, 2H, Ar), 4.91 (s, SCSCH<sub>2</sub>), 2.82 (t, J = 7.6 Hz, CH<sub>2</sub>CON), 1.76 (quin, J = 7.6 Hz,  $-CH_2-CH_2-CH_2-$ ), 1.38–1.25 (m, 28H,  $-CH_2-$ ), 0.88 (t, J = 7.2 Hz,  $-CH_3$ ) ppm.

#### Corrosion tests performed for acidic media

Selection of materials and chemicals used here was made according to Ref. [4] and corrosion tests procedures were also performed according to this Ref. except that the test period and temperature were 25 h and 30°C. The test conditions and obtained results for the corrosion inhibitors 4a-4c are summarized in Table 1.

#### Corrosion tests performed for mineral oil media

Test media and metal selection in additional corrosion tests were according to Ref. [4] except that the period was 24 h.

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