Journal of Organometallic Chemistry 694 (2009) 179–189

Contents lists available at ScienceDirect

Journal of Organometallic Chemistry

journal homepage: www.elsevier.com/locate/jorganchem

Synthesis and characterization of novel pyridyl/naphthyl/(diphenyl) methylseleno substituted alkanoic acids: X-ray structure of 2-pyridylselenoethanoic acid, 2-naphthylselenoethanoic acid and 2-(diphenyl)methylselenoethanoic acid

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ARTICLE INFO

Article history:
Received 1 August 2008
Received in revised form 7 October 2008
Accepted 13 October 2008
Available online 22 October 2008

Keywords:
Ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates
Pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids
β-Lactam
X-ray crystal structure

ABSTRACT

A number of novel and synthetically important pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids (**20**–**25**) have been synthesized using an efficient and operationally simple strategy. Starting substrates, ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates (**8**–**13**) were easily prepared by treatment of ethyl chloroalkanoates **7**(**a**–**c**) with nucleophilic selenium reagent $RSeNa^+$, generated from the cleavage of dipyridyl/dinaphthyl/bis(diphenylmethyl) diselenide (**1**–**6**) with sodium borohydride in ethanol. The ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates (**8**–**13**) on basic hydrolysis and subsequent acidification afford pyridyl/naphthyl/(diphenyl) methylseleno substituted alkanoic acids (**20**–**25**) in excellent yields. These selenoalkanoates (**8**–**13**) and selenoalkanoic acids (**20**–**25**) have been characterized by elemental analysis and various spectroscopic techniques viz. NMR (^1H, ^{13}C and ^{77}Se), IR and mass spectrometry. The molecular structure of 2-pyridylselenoethanoic acid (**20a**), 2-naphthylselenoethanoic acid (**23a**) and 2-(diphenyl)methylselenoethanoic acid (**24a**) has also been established with the help of single crystal X-ray analysis.

1. Introduction

After the discovery of Ebselen [1], organoselenium compounds have emerged as an important class of heterocycles. The past two decades have witnessed a remarkable growth in the field of organoselenium chemistry due to its pivotal role in the synthesis of a large number of biological compounds like selenocarbohydrates, selenoaminoacids and selenopeptides [2–6]. Besides this, organoselenium compounds have showed a great potential for applications in biological systems [7], organic synthesis [8], pharmaceutics [9–10], semiconducting materials [11–13], ligand chemistry [14], biochemistry [15–16] and spectroscopic studies [17].

β-Lactam ring containing compounds are a group of antibiotics of unparalleled importance in chemotherapy. The discoveries of new biologically active β-lactams such as cholesterol acyl transferase inhibitors [18–20], thrombin inhibitors [21], human cytomegalovirus protease inhibitors [22], matrix-metallo protease inhibitors [23], inhibitors of human leukocyte elastase [24–25] and cysteine protease [26–27] and apoptosis inductors [28–29] have provided the motivation for continuous development of new β-lactam systems. Recently, much attention has been devoted towards the synthesis of seleno-β-lactams due to the discovery of novel selenium azetidin-2-ones, such as selenopenams and selenocephams, exhibiting antibacterial activities [30] and β-lactamase inhibitory properties [31].

In persistence to our earlier studies aimed towards the synthesis of novel β-lactams and their functionalization [32–38], we have recently reported the facile synthesis of novel monocyclic 3-phenyl/benzylseleno substituted β-lactams and spiro seleno-β-lactams (Fig. 1) [39] from appropriate synthons 2-phenyl/benzylselenoethanoic acids, which were easily prepared from ethyl phenyl/benzyl selenoethanoates by using an efficient and operationally simple strategy [40].

Further, the biological evaluation of these novel monocyclic 3-phenyl/benzylseleno substituted β-lactams and spiro seleno-β-lactams is underway. In addition, successful attempts have also been made towards the design, synthesis and structural studies of symmetrical and unsymmetrical organochalcogens in our laboratory [41–46].

The biological activity of the particular β-lactam ring is influenced by the type of substitution attached to the basic nucleus [18–31]. So, keeping in view the importance of relationship between biological activity and structural diversity as an essential component, we envisaged to extend above reported studies to

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doi:10.1016/j.jorganchem.2008.10.020

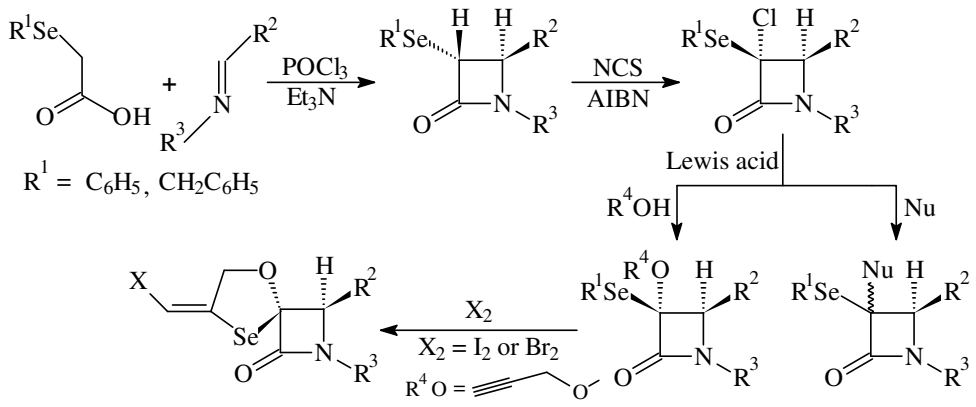


Fig. 1. Synthesis of novel monocyclic 3-phenyl/benzylseleno substituted β-lactams and spiro seleno-β-lactams.

the synthesis of 3-pyridyl/naphthyl/(diphenyl)methylseleno substituted β-lactams and get their biological evaluation. Thus, it was considered of interest to synthesize wide range of structurally diverse pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids, which could serve as synthons for synthesis of 3-pyridyl/naphthyl/(diphenyl)methylseleno substituted β-lactams.

Additionally, seleno substituted alkanoates and alkanoic acids bearing carbonyl functionality will cornerstone to diverse modification of the parent compound. Perin et al. [47] has used aryl selenoalkanoates for the synthesis of highly functionalized vinyl selenides, which in turn can undergo several transformations like [2 + 2] cycloaddition reactions, Diels Alder reactions and serve as Michael acceptors. Clive et al. [48] has reported that 3-(phenylseleno)propanoate can be easily converted by anionic reactions into substrates that undergo sequential ring closing metathesis and radical cyclizations affording bicyclic products. Moreover, employing the ethyl pyridyl/naphthyl/(diphenyl) methylseleno substituted alkanoates and pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids, as reagents would allow installation of sensitive moieties/structural motifs in a variety of compounds in a convenient way.

In this regards, we present here the synthesis, characterization and X-ray structures of variety of structurally diverse ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates and pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids by employing facile synthetic route.

2. Results and discussion

*2.1. Synthesis of ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates **8–13**(**a–c**) and pyridyl/naphthyl/(diphenyl)-methylseleno substituted alkanoic acids **20–25**(**a–c**)*

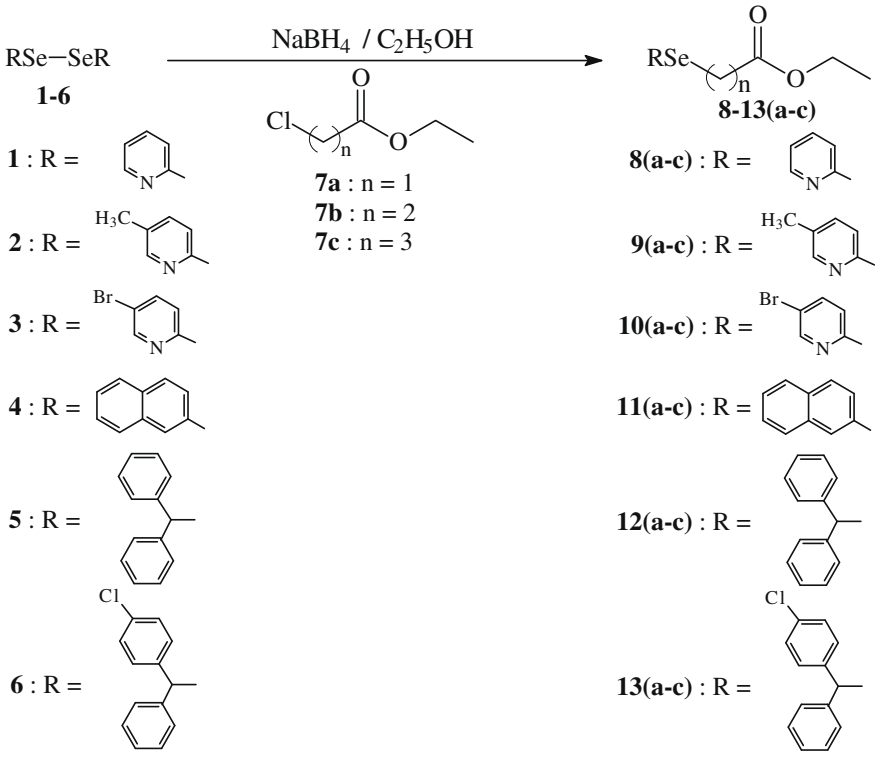
It is an acknowledged fact that the presence of nitrogen in the aromatic ring brings remarkable changes in the properties of the organic compound. Pyridyl/substituted pyridyl organoselenium compounds have emerged as important synthons in heterocylic chemistry, biochemistry, industrial chemistry and coordination chemistry [49–51]. An encouraging report on anti HIV-1 activity of 5-ethyl-6-pyridylthio/seleno acyclouracil further adds new dimensions in pyridyl chalcogens chemistry [52–53]. The naphthyl moiety have also attracted considerable interest due to many attractive features which enables them to be used in asymmetric synthesis, resolution of optically active compounds, synthesis of organic conductors, antioxidants, catalysts and promising ligands in coordination chemistry [54–58].

In literature, a single report has been listed for the synthesis of 2-pyridylselenoethanoic acid by Mautner et al. [59]. The reaction involves the treatment of 2-selenopyridine with chloroacetic acid and sodium bicarbonate in water under reflux conditions followed by neutralization with sodium hydroxide solution and acidification with glacial acetic acid. The resultant product was obtained in 36.6% yield and confirmed by CHN analysis only. Whereas, Fredga [60] has prepared naphthylmethylseleno substituted alkanoic acids form diselenide-dicarboxylic acid, 1-naphthylmethyl chloride or 2-naphthylmethyl bromide, rongalite and ammonia. As a drawback, these synthetic protocols elaborated above suffer from low to moderate yields and lengthy synthetic steps. To the best of our knowledge no report for ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates and pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids have been appeared in literature so far except 2-pyridylselenoethanoic acid.

In our earlier publication [39], we have developed an efficient synthetic route to spiro seleno-β-lactams using *cis*-3-chloro-3-benzylseleno-β-lactams and *cis*-3-alkoxy-3-benzylseleno-β-lactams as the suitable substrates. However, the use of benzylseleno moiety in this reaction scheme afforded the substrates in very low yield. So, it was considered of interest to use (diphenyl)methylseleno moiety instead of dibenzylseleno moiety and would enhance the yield of the (diphenyl)methylseleno substituted β-lactam substrates and spiro seleno-β-lactams.

We have previously reported an efficient method for the synthesis of ethyl phenyl/benzyl selenoalkanoates and phenyl/benzylselenoalkanoic acids [40]. Here, we wish to extend this synthetic approach to synthesize ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates and pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids.

Starting substrates, dipyridyl/dinaphthyl/bis(diphenylmethyl) diselenide (**1–6**) were prepared using the reported procedure [41–46]. Commercially available reagents such as ethyl 2-chloroethanoate **7a,** ethyl 3-chloropropanoate **7b** and ethyl 4-chlorobutanoate **7c** were used as such without further purification. Initially, dipyridyl diselenide (**1a**) was subjected to reductive cleavage by sodium borohydride in ethanolic solution and followed by treatment with 2-chloroethanoate (**7a**) at −5 °C tempearure for 2 min. Progress of the reaction was monitored by thin-layer chromatography (TLC). Purification of the mixture by column chromatography on silica gel using ethyl acetate–hexane (1:99) as the eluant afford 95% yield of ethyl 2-pyridylselenoethanoate (**8a**) as a yellow oil (Scheme 1 and Table 1, entry 1). The **8a** was hydrolyzed with KOH in methanol to produce precipitates of potassium 2-pyridylselenoethanoate (**14a**), which was further acidified with conc. hydrochloric acid to furnish 2-pyridylselenoethanoic acid (**20a**) as a crystalline solid in 90% yield (Scheme 2 and Table 2, entry 1).



Scheme 1. Synthesis of ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates **8–13**(**a–c**).

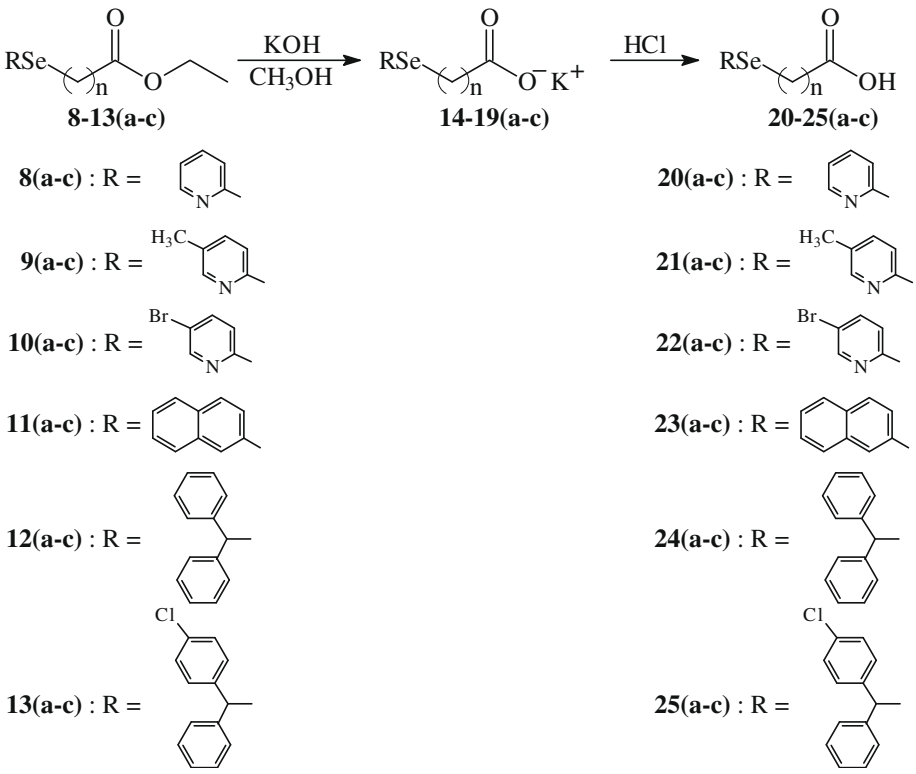
Table 1
Synthesis of ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates **8–13(a–c)**.

Entry	R_2Se_2	Reagent (**7**)	Product	Temperature (°C)	Time (min)	Yield[a] (%)
1	**1**	**7a**	**8a**	−5	2	95
2	**1**	**7b**	**8b**	0–10	10	89
3	**1**	**7c**	**8c**	10–20	30	86
4	**2**	**7a**	**9a**	−5	2	97
5	**2**	**7b**	**9b**	0–15	7	92
6	**2**	**7c**	**9c**	10–20	15	90
7	**3**	**7a**	**10a**	0–5	8	82
8	**3**	**7b**	**10b**	20–25	18	79
9	**3**	**7c**	**10c**	30–45	35	72
10	**4**	**7a**	**11a**	−5–0	10	94
11	**4**	**7b**	**11b**	10–15	30	81
12	**4**	**7c**	**11c**	30–35	85	86
13	**5**	**7a**	**12a**	0–5	5	92
14	**5**	**7b**	**12b**	5–10	12	90
15	**5**	**7c**	**12c**	15–20	25	87
16	**6**	**7a**	**13a**	0–10	10	89
17	**6**	**7b**	**13b**	10–20	20	84
18	**6**	**7c**	**13c**	30–40	60	82

[a] Yields quoted are for isolated products characterized by FT-IR, NMR (^{1}H, ^{13}C, ^{77}Se) and MS.

Similarly, this synthetic approach was followed for different active substrates to obtain ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates **8**(**b–c**), **9–13**(**a–c**) and pyridyl/naphthyl/diphenylmethylseleno substituted alkanoic acids **20**(**b–c**), **21–25**(**a–c**) and the results are summarized in Tables 1 and 2. The selenoalkanoates **8–13**(**a–c**) were obtained as yellow liquids (oils) and could be stored at room temperature for shorter period but preferentially be stored at 0–5 °C temperature conditions for longer period. The resulting compounds are air and moisture stable and easily soluble in polar solvents such as dichloromethane, chloroform and ethyl acetate. The structures of all these synthesized compounds were established on the basis of elemental analysis and various spectroscopic techniques viz., FT-IR, NMR (^{1}H, ^{13}C, ^{77}Se) and mass spectrometry. Further, the molecular structures of 2-pyridylselenoethanoic acid (**20a**), 2-naphthylselenoethanoic acid (**23a**) and 2-(diphenyl)methylselenoethanoic acid (**24a**) have also been established with the help of single crystal X-ray analysis (Fig. 2).

To investigate the role of substituents on product formation, different substrates having electron withdrawing groups or electron releasing groups were used. As presented in Table 1, the presence of electron withdrawing group on substrates 3, increases the reaction temperature conditions, enhances the reaction time and lowers the yield of the desired selenoalkanoates **10(a–c)** in comparison to substrate 2 possessing electron releasing group (Table 1, entry 4–9). The plausible reason for this may be based on the stability of RSe^-Na^+ as depicted in mechanistic pathway of the reaction shown in Scheme 3.



Scheme 2. Synthesis of pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids **20–25**(**a–c**).

Table 2
Pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids **20–25(a–c)**.

Entry	Product	Yield[a] (%)	M.p. (°C)
1	**20a**	90	120–122
2	**20b**	88	60–61
3	**20c**	81	40–42
4	**21a**	92	57–59
5	**21b**	91	49–51
6	**21c**	89	Oil
7	**22a**	76	96–97
8	**22b**	74	92–93
9	**22c**	71	Semisolid
10	**23a**	89	39–40
11	**23b**	83	40–42
12	**23c**	77	43–44
13	**24a**	87	96–99
14	**24b**	86	88–89
15	**24c**	81	Semisolid
16	**25a**	85	85–87
17	**25b**	79	79–80
18	**25c**	75	Semisolid

[a] Yields quoted are for isolated products characterized by FT-IR, NMR (^{1}H, ^{13}C, ^{77}Se) and MS.

The naked anion $RSeNa^+$ which is weakly coordinated to sodium cation are believed to react efficiently with various chloroalkanoates **7(a–c)** at ambient temperature to afford desired selenoalkanoates **8–13(a–c)**. The presence of electron withdrawing groups or electron releasing groups would enhance or retard the stability of RSe^-Na^+ anion, respectively. When this protocol was employed in THF and methanol instead of ethanol as solvent, the products were obtained in poor yield with incomplete consumption of starting substrates dipyridyl/dinaphthyl/bis(diphenylmethyl) diselenide (**1–6**).

Further elaboration of the pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids (**20a–25a**) to potential monocyclic and spirocyclic seleno-β-lactams is underway in our laboratory. Additionally, this synthetic approach would be explored for the synthesis of tellurium substituted alkanoates and alkanoic acids.

2.2. X-ray crystallographic analysis

The crystal structures of 2-pyridylselenoethanoic acid (**20a**), 2-naphthylselenoethanoic acid (**23a**) and 2-(diphenyl)methylselenoethanoic acid (**24a**) were confirmed by X-ray crystallographic analysis (Fig. 2). The acids **20a**, **23a** and **24a** were crystallized from dichloromethane–hexane (3:1) as colorless crystalline solids suitable for single crystal X-ray diffraction. A prospective view of the molecular structure with atom numbering scheme has been given in Fig. 2. Selected bond lengths [Å] and bond angles [°] are presented in Table 3. All the relevant information about data collection and refinement parameters has been listed in Table 4.

The Se–C bond length in **20a** [Se–C(2), 1.94 Å], **23a** [Se–C(11), 1.95 Å], and **24a**[Se–C(14), 1.95 Å] lie within the range (1.90–1.95 Å), reported for several aliphatic selenocompounds possessing Se–C bond [61–62]. The bond angle of C–Se–C bond in **20a**, **23a** and **24a** are 99.67(9), 99.4(3) and 98.59(9), respectively. This bond angles indicate the distortion of sp^3 carbon from its regular tetrahedral geometry and established the 'V' shaped geometry about C–Se–C bond. The hydrogen bonding interactions have been observed with in a crystal lattice of these compounds. Interestingly, the 2-pyridylselenoethanoic acid (**20a**) has shown the hydrogen bonding interactions between the nitrogen atom of pyridyl ring of one crystal unit and hydrogen atom of the carboxyl group of other crystal unit. However, the 2-naphthylselenoethanoic acid (**23a**) and 2-(diphenyl)methylseleno ethanoic acid (**24a**) depicted these interactions with carbonyl oxygen of carboxyl group of first crystal unit with hydrogen atom of the carboxyl group of second crystal unit. Compound **23a** has relatively high residuals in the Fourier map or final difference map and were located near the Se atom. This may be due to absorption effects.

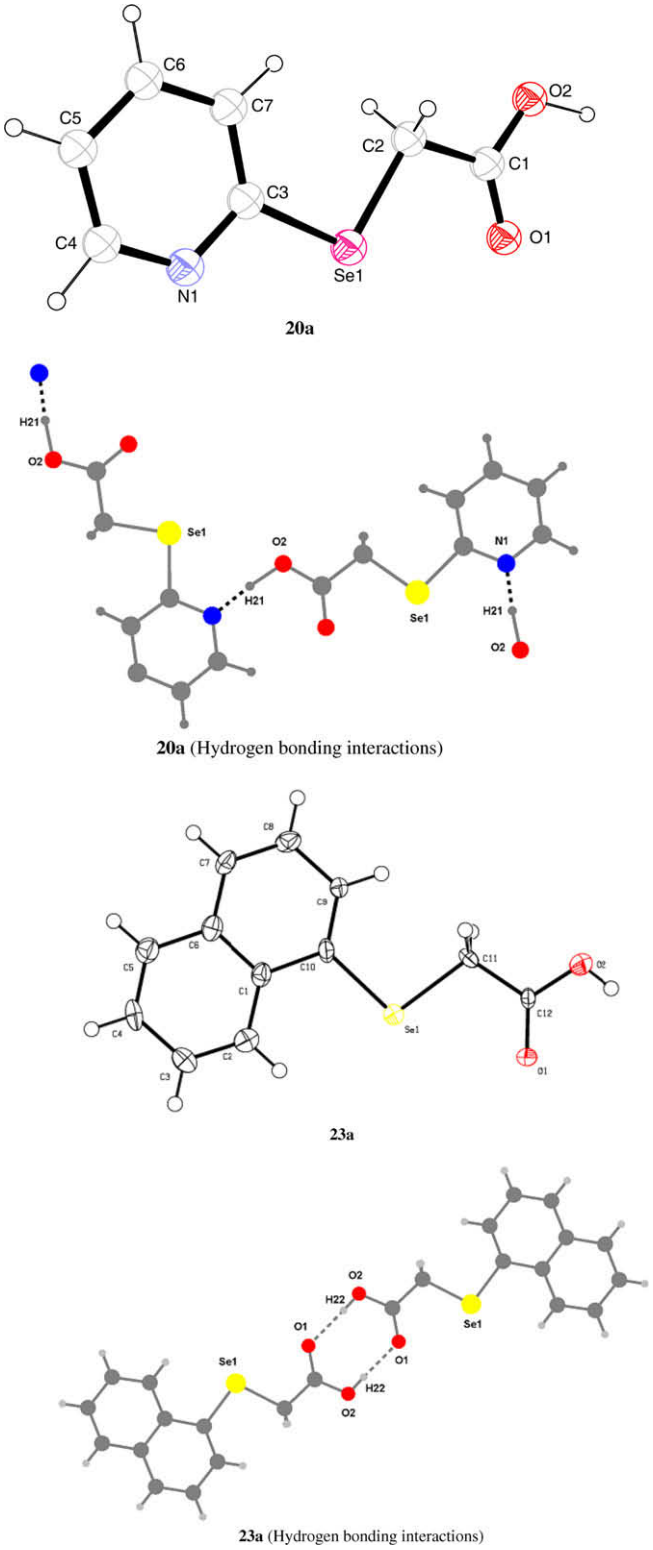


Fig. 2. ORTEP diagrams of 2-pyridylselenoethanoic acid (**20a**), 2-naphthylselenoethanoic acid (**23a**) and 2-(diphenyl)methylselenoethanoic acid (**24a**), showing atom numbering and hydrogen bonding interactions.

2.3. Spectroscopic studies

FT-IR, NMR (^{1}H, ^{13}C, ^{77}Se) spectroscopy and mass spectrometry (only in few representative cases) supported the formulation of the target compounds. Initially, the addition of –CH_2–COO–functionality to seleno substituted group in the selenoalkanoates **8–13**(**a–c**) was confirmed from the IR spectra, which showed absorption band in the range of 1708–1740 cm^{-1} (C=O). However, in some cases, after work up the products were identified as the starting substrates only, dipyridyl/dinaphthyl/bis(diphenylmethyl) diselenide (**1–6**) as evident from the absence of C=O absorption band in IR spectra. It may be due to the scission of Se–C(H_2) bond at higher temperature and generation of the RSe free radical which gives diselenide (**1–6**).

The ^{1}H, ^{13}C and ^{77}Se NMR data of selenoalkanoates **8–13**(**a–c**) and selenoalkonic acids **20–25**(**a–c**) were obtained as expected and listed in the experimental section. The ^{1}H, ^{13}C and ^{77}Se NMR results for seleno substituted methine protons (RSeCH_2COO–) of selenoethanoates **8a–13a** and selenoethanoic acids **20a–25a** were tabulated in the Table 5 for comparative study. These results shows upfield shift of 0.18–1.16 ppm (^{1}H NMR) from pyridyl to naphthyl moiety and further to (diphenyl)methyl group. This study postulates the greater deshielding influence of electron withdrawing pyridine ring over naphthyl and (diphenyl)methyl group successively. ^{13}C and ^{77}Se NMR spectroscopic results also reveal a similar trend. In addition, the evaluation of ^{77}Se NMR spectra of compounds **8–13**(**a–c**), **20–25**(**a–c**) showed the decrease in chemical shift as the number of intervening methylene groups increases, which also correlates with our earlier studies [41,43,63].

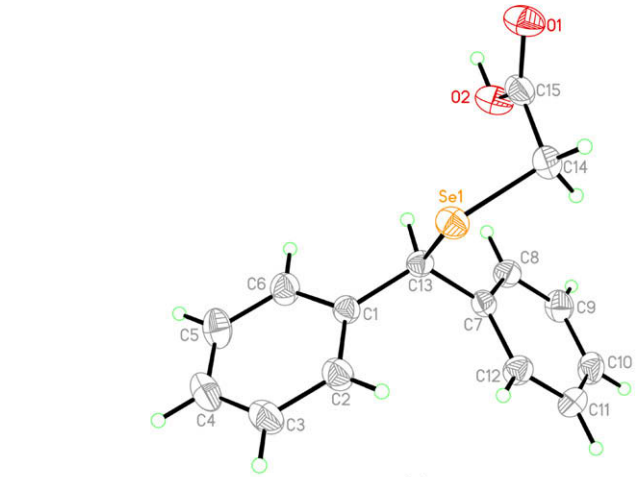
The mass spectrum (EI-MS) of these selenoalkanoates and selenoalkanoic acids was found to be complicated due to natural abundance of six stable isotopes of selenium. In the mass spectra of ethyl 2-pyridylselenoethanoate (**8a**) and 2-pyridylselenoethanoic acid (**20a**), the base peak does not correspond to molecular ion peak and appears at *m/z* 246 (100%), 218 (100%) for [M+2] ion, while, the spectra display the molecular ion peak [M]$^+$ at *m/z* 244 (56%), 216 (46%), respectively. However, the fragmentation pattern of ethyl 2-pyridylselenobutanoate (**8c**) and ethyl 2-(5-bromo)pyridylselenoethanoate (**10a**) exhibit the resemblance of molecular ion peak and base peak at *m/z* 272 (100%), 323 (100%), respectively. In the EI-MS spectra of ethyl 2-naphthylselenoethanoate (**11a**) and 2-naphthylselenoethanoic acid (**23a**), the base peak was observed at *m/z* 311 (100%), 283 (100%), respectively and corresponds to [M+18] ion, whereas, the molecular ion peak were identified at *m/z* 293 (7%), 283 (14%), with very low intensity. The mass spectrum of ethyl 2-(diphenyl)methylselenobutanoate (**12c**) showed the molecular ion peak at *m/z* 361 (89%) and base peak at *m/z* 363 (100%) for [M+2] ion. The other prominent peak at *m/z* 167 (38%) represent for $(Ph_2CH)^+$ ion.

3. Experimental

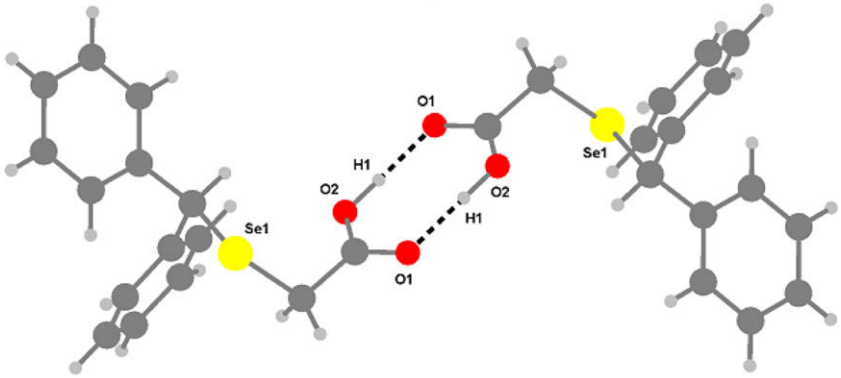
3.1. General

Melting points were determined in an open capillary on melting point apparatus and are uncorrected. Infrared spectra were taken on a Perkin–Elmer 1430 (FTIR) spectrophotometer (υ_{max} in cm^{-1}). ^{1}H (300 MHz), ^{13}C (75 MHz) and ^{77}Se (57 MHz) NMR spectra were recorded on JEOL AL 300 (300 MHz) spectrometer. Chemical shifts are given in ppm relative to Me_4Si as an internal standard (δ = 0 ppm) for ^{1}H NMR, $CDCl_3$ (δ = 77.0 ppm) for ^{13}C NMR spectra and Me_2Se (δ = 0 ppm) for ^{77}Se NMR spectra. The mass spectra (EI) were obtained using QTOF mass spectrometer. The elemental analysis (C, H, N) were recorded on Flash EA 112 elemental analyzer. Column chromatography was performed using Merck Silica Gel (60–120 mesh) using ethyl acetate–hexanes (1:99) as an eluant system. Analytical thin-layer chromatography (TLC) was performed using Merck Silica Gel G using ethyl acetate–hexanes (2:98) as an eluant system. For visualization, TLC plates were stained with iodine vapors.

The reactions for the preparation of selenoalkanoates **8–13** (**a–c**) were carried out under dry and deoxygenated nitrogen atmosphere. Sodium borohydride (Qualigen), ethyl 2-chloroethan-



24a



24a (Hydrogen bonding interactions)

Fig. 2 *(continued)*

oate (AIFA AESAR), ethyl 3-chloropropanoate (AIFA AESAR) and ethyl 4-chlorobutanoate (MERCK), absolute alcohol (TEDIA) and all other commercially available compounds/reagents were of analytical grade and used without further purification. THF was distilled under N_2 from sodium-benzophenone immediately before use.

3.2. Crystal structure analysis

Diffraction quality, colorless single crystals of compound **20a, 23a** and **24a**, undertaken for crystallographic study were grown from the slow evaporation of dichloromethane–hexane (3:1) solution of the compounds. Single crystals were mounted on glass capillaries of Bruker Smart Apex diffractometer using graphite-monochromated Mo Kα radiation at room temperature. The data integration and reduction were processed with SAINT software. The crystal structure of these compounds were solved by direct methods using SHELX-97 [64] and refined by full-matrix least squares method. All the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were included in the ideal position with fixed isotropic U values and were riding. The empirical absorption corrections for these compounds were performed using SADBAS program [65]. All other information regarding the refinement is given in Table 4 and the ORTEP representations are with atom numbering scheme (thermal ellipsoids are at 50% probability level) (Fig. 2).

3.3. Synthesis of ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates ***8–13(a–c)***

The ethyl pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoates **8–13**(**a–c**) were synthesized by the procedure as reported earlier for the preparation of phenyl/benzylseleno substituted alkanoates [40] in the cited reference, however, the reaction temperature and completion time was varied depending upon the nature of the starting substrates, dipyridyl/dinaphthyl/bis (diphenylmethyl) diselenide (**1–6**).

3.3.1. Ethyl 2-pyridylselenoethanoate [$(C_5H_4N)SeCH_2COOC_2H_5$](***8a***)

Yield 95%; yellow oil; ^{1}H NMR: δ 8.43–8.45 (d, 1H, 4.0 Hz), 7.45–7.50 (t, 1H, 7.4 Hz), 7.37–7.39 (d, 1H, 8.0 Hz), 7.03–7.06 (t, 1H, 7.2 Hz), 4.16–4.20 (q, 2H, 7.1 Hz), 3.88 (s, 2H), 1.22–1.25 (t, 3H, 7.1 Hz); ^{13}C NMR: δ 170.95, 153.81, 150.10, 136.33, 125.13, 120.85, 61.56, 25.43, 14.17; ^{77}Se NMR: δ 362.09; IR ($CHCl_3$, ν cm^{-1}): 1732.6; MS–EI, *m/e* (R.I., assignment): 246 (100, $[M+2]^+$), 244 (56, $[M]^+$).

$$RSe{-}SeR \xrightarrow{NaBH_4 / C_2H_5OH} \left[RSe^{-}Na^{+}\right] \xrightarrow[n = 1, 2, 3]{\overset{-}{Cl}(\overset{+}{C}H_2)_nCOOC_2H_5} RSe(CH_2)_nCOOC_2H_5$$

Scheme 3. Plausible reaction pathway for the formation of ethyl pyridyl/naphthyl/(diphenyl)methyl seleno substituted alkanoates **8–13**(**a–c**).

Table 3
Selected bond parameters of **20a**, **23a** and **24a**.

Bond length (Å)	Bond angle (°)		
*Compound (**20a**)*			
Se(1)–C(3)	1.907(2)	C(3)–Se(1)–C(2)	99.67(9)
Se(1)–C(2)	1.940(2)	C(4)–N(1)–(C3)	119.31(18)
O(1)–C(1)	1.227(3)	O(1)–C(1)–O(2)	125.2(2)
N(1)–C(4)	1.303(3)	O(1)–C(1)–C(2)	122.06(19)
N(1)–C(3)	1.346(3)	O(2)–C(1)–C(2)	112.76(18)
C(1)–O(2)	1.348(3)	C(1)–C(2)–Se(1)	107.29(14)
C(1)–C(2)	1.303(3)	N(1)–C(3)–C(7)	121.61(19)
C(3)–C(7)	1.516(3)	N(1)–C(3)–Se(1)	112.93(14)
C(4)–C(5)	1.393(3)	C(7)–C(3)–Se(1)	125.46(16)
C(5)–C(6)	1.378(3)	N(1)–C(4)–C(5)	122.3(2)
C(6)–C(7)	1.384(3)		
*Compound (**23a**)*			
Se(1)–C(10)	1.930(7)	C(10)–Se(1)–C(11)	99.4(3)
Se(1)–C(11)	1.952(6)	O(1)–C(12)–O(2)	123.9(6)
O(2)–C(12)	1.303(7)	O(1)–C(12)–C(11)	123.0(5)
O(1)–C(12)	1.229(8)	O(2)–C(12)–C(11)	113.1(5)
C(4)–C(5)	1.372(11)	C(9)–C(10)–Se(1)	122.4(5)
C(4)–C(3)	1.405(10)	C(1)–C(10)–Se(1)	116.1(5)
C(6)–C(7)	1.419(10)	C(12)–C(11)–Se(1)	109.1(4)
C(6)–C(5)	1.422(10)		
C(6)–C(1)	1.424(9)		
C(12)–C(11)	1.491(8)		
C(7)–C(8)	1.371(10)		
C(2)–C(3)	1.372(10)		
C(2)–C(1)	1.398(10)		
C(10)–C(9)	1.356(9)		
C(10)–C(1)	1.442(9)		
C(8)–C(9)	1.427(9)		
*Compound (**24a**)*			
C(13)–Se(1)	1.982(2)	C(7)–C(13)–C(1)	116.63(16)
C(14)–C(15)	1.486(3)	C(7)–C(13)–Se(1)	111.92(12)
C(14)–Se(1)	1.951(2)	C(1)–C(13)–Se(1)	106.01(12)
C(14)–H(14)A	0.9700	Se(1)–C(13)–H(13)	107.3
C(14)–H(14)B	0.9700	C(15)–C(14)–Se(1)	109.85(14)
C(15)–O(2)	1.260(3)	Se(1)–C(14)–H(14A)	109.7
C(15)–O(1)	1.265(3)	Se(1)–C(14)–H(14B)	109.7
O(2)–H(1)	0.99(5)	H(14A)–C(14)–H(14B)	108.2
C(1)–C(6)	1.380(3)	O(2)–C(15)–O(1)	122.6(2)
C(1)–C(2)	1.382(3)	O(2)–C(15)–C(14)	118.7(2)
C(1)–C(13)	1.517(3)	O(1)–C(15)–C(14)	118.7(2)
C(7)–C(13)	1.511(3)	C(15)–O(2)–H(1)	122(3)
		C(14)–Se(1)–C(13)	98.59(9)

Table 4
Crystallographic data and measurements of compounds **20a**, **23a** and **24a**.

	Compound **20a**	Compound **23a**	Compound **24a**
Empirical formula	$C_7H_7NO_2Se$	$C_{12}H_{10}O_2Se$	$C_{15}H_{14}O_2Se$
Formula weight (g/mol)	216.10	265.16	305.23
Temperature (K)	150(2)	293(2)	298(2)
Diffractometer used	Bruker Smart Apex	Bruker Smart Apex	Bruker Smart Apex
Radiation used, λ Mo Kα (Å)	0.71073	0.71069	0.71073
Crystal system/ space group	Orthorhombic, P_{nma}	Triclinic, $P\bar{1}$	Monoclinic, $P2_{1/c}$
Unit cell dimensions			
a (Å)	14.7343(7)	7.191(5)	15.322(5)
b (Å)	6.5757(3)	7.739(5)	10.553(3)
c (Å)	7.8016(4)	10.384(5)	8.361(3)
α (°)	90.00	105.279(5)	90.00
β (°)	90.00	108.626(5)	93.131(5)
γ (°)	90.00	92.536(5)	90.00
V (Å^3)	755.88(6)	523.0(6)	1349.8(7)
Z, calculated density (mg/m^3)	4	2	2
Absorption coefficient (mm^{-1})	4.911	3.564	2.773
F(000)	424	264	616
Crystal size (mm^3)	0.70 × 0.20 × 0.20	0.14 × 0.12 × 0.10	0.36 × 0.26 × 0.15
θ range for data collection (°)	2.76–29.91	2.76–28.31	2.34–25.98
Index ranges	$-20 \leqslant h \leqslant 20$ $-7 \leqslant k \leqslant 8$ $-10 \leqslant l \leqslant 8$	$-8 \leqslant h \leqslant 9$ $-9 \leqslant k \leqslant 10$ $-13 \leqslant l \leqslant 10$	$-18 \leqslant h \leqslant 18$ $-12 \leqslant k \leqslant 12$ $-10 \leqslant l \leqslant 10$
Reflection collected/unique	9793/1154	3367/2451	13503/2642
[$R_{(int)}$]	0.0232	0.0167	0.0265
Reflection with [$I > 2\sigma(I)$]	1002	2094	2286
Refinement method	Full-matrix least-square on F^2	Full-matrix least-square on F^2	Full-matrix least-square on F^2
Final R indices, [$I > 2\sigma(I)$]	R_1 = 0.0218, wR_2 = 0.0519	R_1 = 0.0531, wR_2 = 0.1206	R_1 = 0.0260, wR_2 = 0.0658
R indices [all data]	R_1 = 0.0219, wR_2 = 0.0544	R_1 = 0.0773, wR_2 = 0.1808	R_1 = 0.0313, wR_2 = 0.0681
Largest difference in peak and hole (e Å^{-3})	0.583 and −0. 331	1.654 and −1.847	0.214 and −0.406

*3.3.2. Ethyl 2-pyridylselenopropanoate [$(C_5H_4N)Se(CH_2)_2COOC_2H_5$] (**8b**)*

Yield 89%; yellow oil; ^{1}H NMR: δ 8.32–8.34 (d, 1H, 4.8 Hz), 7.28–7.33 (t, 1H, 7.5 Hz), 7.15–7.20 (d, 1H, 4.8 Hz), 6.87–6.92 (t, 1H, 7.2 Hz), 4.07–4.10 (q, 2H, 7.1 Hz), 3.26–3.31 (t, 2H, 7.1 Hz), 2.75–2.79 (t, 2H, 6.9 Hz), 1.17–1.22 (t, 3H, 7.1 Hz); ^{13}C NMR: δ 171.77, 155.14, 150.02, 135.47, 125.29, 119.90, 60.22, 35.47, 19.38, 14.17; ^{77}Se NMR: δ 357.67; IR ($CHCl_3$, ν cm^{-1}): 1735.6.

*3.3.3. Ethyl 2-pyridylselenobutanoate [$(C_5H_4N)Se(CH_2)_3COOC_2H_5$] (**8c**)*

Yield 86%; yellow oil; ^{1}H NMR: δ 8.42–8.44 (d, 1H, 4.8 Hz), 7.41–7.46 (t, 1H, 7.9 Hz), 7.31–7.33 (d, 1H, 8 Hz), 7.00–7.03 (t, 1H, 4.8 Hz), 4.10–4.15 (q, 2H, 7.1 Hz), 3.20–3.24 (t, 2H, 7.1 Hz), 2.45–2.49 (t, 2H, 7.2 Hz), 2.08–2.15 (m, 2H), 1.23–1.27 (t, 3H, 7.1 Hz); ^{13}C NMR: δ 173.18, 155.11, 150.13, 135.99, 125.51, 120.35, 60.47, 34.24, 25.72, 24.90, 14.31; ^{77}Se NMR: δ 344.21; IR ($CHCl_3$, ν cm^{-1}): 1735.7; MS-EI, *m/e* (R.I., assignment): 272 (100, [M]$^+$), 270 (42, [$C_5H_4NSe(CH_2)_3COOCH_2CH$]$^+$).

*3.3.4. Ethyl 2-(5-methyl)pyridylselenoethanoate [(5-$CH_3C_5H_3N$)Se-$CH_2COOC_2H_5$] (**9a**)*

Yield 97%; yellow oil; ^{1}H NMR: δ 8.18 (s, 1H), 7.14–7.22 (m, 2H), 4.03–4.10 (q, 2H, 7.2 Hz), 3.78 (s, 2H), 2.20 (s, 3H), 1.14–1.19 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 170.33, 150.21, 149.95, 136.83, 129.87, 124.43, 60.99, 25.04, 17.83, 14.13; ^{77}Se NMR: δ 355.18; IR ($CHCl_3$, ν cm^{-1}): 1733.0.

Table 5
^{1}H, ^{13}C and ^{77}Se NMR values of seleno substituted methine protons (–SeCH_2COO–) in selenoethanoates **8a–13a** and selenoethanoic acids **20a–25a**.

Entry	Compound		δ ^{1}H NMR	δ ^{13}C NMR	δ ^{77}Se NMR
	Selenoethanoates				
1	$(C_5H_4N)SeCH_2COOC_2H_5$	**8a**	3.98	25.43	362.09
2	(5-$CH_3C_5H_3N$)Se$CH_2COOC_2H_5$	**9a**	3.78	25.04	335.18
3	(5-BrC_5H_3N)Se$CH_2COOC_2H_5$	**10a**	3.80	25.39	358.71
4	(1-$C_{10}H_7$)Se$CH_2COOC_2H_5$	**11a**	3.41	27.65	269.19
5	$(C_6H_5)_2CHSeCH_2COOC_2H_5$	**12a**	2.85	28.10	248.15
6	(4-ClC_6H_4)(C_6H_5)CHSeCH_2COO C_2H_5	**13a**	2.82	24.41	244.86
	Selenoethanoic acids				
7	$(C_5H_4N)SeCH_2COOH$	**20a**	3.67	26.78	391.28
8	(5-$CH_3C_5H_3N$)SeCH_2COOH	**21a**	3.51	26.35	371.45
9	(5-BrC_5H_3N)SeCH_2COOH	**22a**	3.65	26.59	380.04
10	(1-$C_{10}H_7$)SeCH_2COOH	**23a**	3.48	27.21	275.14
11	$(C_6H_5)_2CHSeCH_2COOH$	**24a**	2.88	24.03	247.16
12	(4-ClC_6H_4)(C_6H_5)CHSeCH_2COOH	**25a**	2.81	23.92	241.39

*3.3.5. Ethyl 2-(5-methyl)pyridylselenopropanoate [(5-$CH_3C_5H_3N$)Se(CH_2)$_2$$COOC_2H_5$] (**9b**)*

Yield 92%; yellow oil; ^{1}H NMR: δ 8.03 (s, 1H), 7.01–7.12 (m, 2H), 3.97–4.03 (q, 2H, 7.2 Hz), 3.55–3.61 (t, 2H, 7.1 Hz), 2.54–2.58 (t, 2H, 7.1 Hz), 2.19 (s, 3H), 1.02–1.07 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.27, 150.03, 149.67, 136.34, 129.74, 124.43, 60.59, 34.83, 25.04, 17.64, 14.27; ^{77}Se NMR: δ 331.17; IR ($CHCl_3$, ν cm^{-1}): 1732.1.

*3.3.6. Ethyl 2-(5-methyl)pyridylselenobutanoate [(5-$CH_3C_5H_3N$)Se(CH_2)$_3$$COOC_2H_5$] (**9c**)*

Yield 90%; yellow oil; ^{1}H NMR: δ 8.16 (s, 1H), 7.02–7.16 (m, 2H), 3.99–4.06 (q, 2H, 7.2 Hz), 3.08–3.13 (t, 2H, 6.9 Hz), 2.33–2.38 (t, 2H, 6.9 Hz), 2.18 (s, 3H), 1.94–2.05 (m, 2H), 1.15–1.20 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 172.55, 151.19, 150.28, 136.53, 129.26, 128.95, 128.17, 125.29, 124.92, 60.03, 34.04, 25.74, 24.69, 17.68, 14.33; ^{77}Se NMR: δ 326.41; IR ($CHCl_3$, ν cm^{-1}): 1732.1.

*3.3.7. Ethyl 2-(5-bromo)pyridylselenoethanoate [(5-BrC_5H_3N)$SeCH_2$-$COOC_2H_5$] (**10a**)*

Yield 82%; yellow oil; ^{1}H NMR: δ 8.39 (s, 1H), 7.48–7.55 (d, 1H, 8.4 Hz), 7.15–7.18 (d, 1H, 8.4 Hz), 4.04–4.11 (q, 2H, 6.9 Hz), 3.78 (s, 2H), 1.14–1.19 (t, 3H, 6.9 Hz); ^{13}C NMR: δ 169.76, 151.01, 138.41, 128.02, 125.70, 117.52, 61.15, 25.39, 14.04; ^{77}Se NMR: δ 358.71; IR ($CHCl_3$, ν cm^{-1}): 1732.0; MS-EI, *m/e* (R.I., assignment): 323 (100, $[M]^+$), 321 (49, $[(5\text{-}BrC_5H_3N)SeCH_2COOCH_2CH]^+$), 277 (33, $[(5\text{-}BrC_5H_3N)SeCH_2CO]^+$).

*3.3.8. Ethyl 2-(5-bromo)pyridylselenopropanoate [(5-BrC_5H_3N)Se(CH_2)$_2$$COOC_2H_5$] (**10b**)*

Yield 79%; yellow oil; ^{1}H NMR: δ 8.38 (s, 1H), 7.43–7.51 (d, 1H, 8.4 Hz), 7.13–7.16 (d, 1H, 8.4 Hz), 4.02–4.07 (q, 2H, 7.2 Hz), 3.13–3.19 (t, 2H, 7.2 Hz), 2.40–2.44 (t, 2H, 7.2 Hz), 1.17–1.22 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.03, 150.78, 137.74, 128.72, 125.68, 116.95, 60.38, 36.13, 25.27, 14.13; ^{77}Se NMR: δ 341.25; IR ($CHCl_3$, ν cm^{-1}): 1731.4.

*3.3.9. Ethyl 2-(5-bromo)pyridylselenobutanoate [(5-BrC_5H_3N)Se(CH_2)$_3$$COOC_2H_5$] (**10c**)*

Yield 72%; yellow oil; ^{1}H NMR: δ 8.39 (s, 1H), 7.43–7.47 (d, 1H, 8.4 Hz), 7.11–7.13 (d, 1H, 8.1 Hz), 4.01–4.08 (q, 2H, 7.2 Hz), 3.11–3.15 (t, 2H, 7.2 Hz), 2.33–2.38 (t, 2H, 7.2 Hz), 1.97–2.08 (m, 2H), 1.17–1.22 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 172.06, 150.21, 136.14, 126.02, 125.47, 116.81, 60.11, 36.32, 24.98, 19.98, 14.09; ^{77}Se NMR: δ 328.48; IR ($CHCl_3$, ν cm^{-1}): 1730.7.

*3.3.10. Ethyl 2-naphthylselenoethanoate [(1-$C_{10}H_7$)$SeCH_2COOC_2H_5$] (**11a**)*

Yield 94%; greenish-yellow oil; ^{1}H NMR: δ 8.29–8.32 (d, 1H, 8.4 Hz), 7.82–7.84 (d, 1H, 7.2 Hz), 7.73–7.75 (d, 2H, 8.1 Hz), 7.39–7.51 (m, 2H), 7.27–7.32 (t, 1H, 7.2 Hz), 3.88–3.95 (q, 2H, 7.2 Hz), 3.41 (s, 2H), 0.97–1.02 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.95, 134.76, 133.94, 133.29, 129.03, 128.74, 128.64, 127.77, 126.58, 126.35, 125.63, 60.81, 27.65, 14.08; ^{77}Se NMR: δ 269.19; IR ($CHCl_3$, ν cm^{-1}): 1731.8; MS-EI, *m/e* (R.I., assignment): 311 (100, $[M+18]^+$), 293 (7, $[M]^+$), 292 (40, $[M-1]^+$), 278 (16, $[(1\text{-}C_{10}H_7)SeCH_2\text{-}COOCH_2]^+$.

*3.3.11. Ethyl 2-naphthylselenopropanoate [(1-$C_{10}H_7$)Se(CH_2)$_2$$COOC_2$-$H_5$] (**11b**)*

Yield 81%; greenish-yellow oil; ^{1}H NMR: δ 8.38–8.41 (d, 1H, 8.4 Hz), 7.81–7.83 (d, 1H, 7.4 Hz), 7.78–7.80 (d, 2H, 7.8 Hz), 7.47–7.56 (m, 2H), 7.33–7.37 (t, 1H, 7.1 Hz), 4.05–4.10 (q, 2H, 7.1 Hz), 3.09–3.12 (t, 2H, 7.4 Hz), 2.63–2.67 (t, 2H, 7.4 Hz), 1.18–1.21 (t, 3H, 7.1 Hz); ^{13}C NMR: δ 172.17, 134.48, 134.01, 133.35, 128.83, 128.66, 128.47, 127.74, 126.78, 126.27, 125.75, 60.67, 35.36, 21.96, 14.19; ^{77}Se NMR: δ 253.87; IR ($CHCl_3$, ν cm^{-1}): 1735.8.

*3.3.12. Ethyl 2-naphthylselenobutanoate [(1-$C_{10}H_7$)Se(CH_2)$_3$$COOC_2H_5$] (**11c**)*

Yield 86%; greenish-yellow oil; ^{1}H NMR: δ 8.36–8.38 (d, 1H, 8.2 Hz), 7.81–7.83 (d, 1H, 7.8 Hz), 7.76–7.78 (d, 2H, 7.4 Hz), 7.47–7.56 (m, 2H), 7.34–7.38 (t, 1H, 7.9 Hz), 4.05–4.10 (q, 2H, 7.1 Hz), 2.94–2.98 (t, 2H, 7.2 Hz), 2.40–2.44 (t, 2H, 7.2 Hz), 1.93–2.00 (m, 2H), 1.18–1.22 (t, 3H, 7.1 Hz); ^{13}C NMR: δ 172.96, 134.33, 134.02, 132.36, 129.12, 128.68, 128.36, 127.56, 126.66, 126.23, 125.89, 60.43, 34.05, 27.29, 25.35, 14.23; ^{77}Se NMR: δ 244.12; IR ($CHCl_3$, ν cm^{-1}): 1735.8.

*3.3.13. Ethyl 2-(diphenyl)methylselenoethanoate [(C_6H_5)$_2$$CHSeCH_2$-$COOC_2H_5$] (**12a**)*

Yield 92%; yellow oil; ^{1}H NMR: δ 7.35–7.38 (m, 4H), 7.09–7.23 (m, 6H), 5.58 (s, 1H), 3.99–4.11 (q, 2H, 7.2 Hz), 2.85 (s, 2H), 1.16–1.24 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.11, 140.57, 128.84, 128.57, 127.27, 60.93, 48.62, 24.40, 14.23; ^{77}Se NMR: δ 248.15; IR ($CHCl_3$, ν cm^{-1}): 1730.8.

*3.3.14. Ethyl 2-(diphenyl)methylselenopropanoate [(C_6H_5)$_2$$CHSe(CH_2)_2$-$COOC_2H_5$] (**12b**)*

Yield 90%; yellow oil; ^{1}H NMR: δ 7.32–7.38 (m, 4H), 7.08–7.22 (m, 6H), 5.35 (s, 1H), 4.00–4.07 (q, 2H, 7.2 Hz), 2.44–2.56 (m, 4H), 1.14–1.20 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.88, 141.43, 141.40, 128.93, 128.74, 128.57, 127.10, 60.47, 47.91, 35.12, 19.58, 14.34; ^{77}Se NMR: δ 241.12; IR ($CHCl_3$, ν cm^{-1}): 1735.8.

*3.3.15. Ethyl 2-(diphenyl)methylselenobutanoate [(C_6H_5)$_2$$CHSe(CH_2)_3$-$COOC_2H_5$] (**12c**)*

Yield 86%; yellow oil; ^{1}H NMR:: δ 7.25–7.30 (m, 4H), 7.15–7.18 (m, 6H), 5.43 (s, 1H), 4.01–4.08 (q, 2H, 7.2 Hz), 2.31–2.35 (t, 2H, 7.2 Hz), 2.22–2.26 (t, 2H, 7.2 Hz), 1.80–1.85 (m, 2H), 1.15–1.18 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.46, 141.27, 128.88, 128.60, 128.54, 127.01, 60.35, 47.55, 35.08, 21.71, 19.22, 14.19; ^{77}Se NMR: δ 238.11; IR ($CHCl_3$, ν cm^{-1}): 1732.0; MS-EI, *m/e* (R.I., assignment): 362 (100, $[M+1]^+$), 361 (89, $[M]^+$), 316 (84, $[(C_6H_5)_2CHSe(CH_2)_3\text{-}CO]^+$), 167 (38, $[(C_6H_5)_2CH]^+$).

*3.3.16. Ethyl 2-[(4-chlorophenyl)(phenyl)]methylselenoethanoate [(4-ClC_6H_4)(C_6H_5)$CHSeCH_2COO$ C_2H_5] (**13a**)*

Yield 89%; yellow oil; ^{1}H NMR: δ 7.30–7.34 (m, 4H), 7.11–7.24 (m, 5H), 5.56 (s, 1H), 4.02–4.09 (q, 2H, 6.9 Hz), 2.82 (s, 2H), 1.19–1.23 (t, 3H, 6.9 Hz); ^{13}C NMR: δ 170.88, 140.17, 139.27, 133.27, 130.29, 128.84, 128.78, 127.57, 61.02, 47.81, 24.41, 14.34; ^{77}Se NMR: δ 244.86; IR ($CHCl_3$, ν cm^{-1}): 1728.4.

*3.3.17. Ethyl 2-[(4-chlorophenyl)(phenyl)]methylselenopropanoate [(4-ClC_6H_4)(C_6H_5)$CHSe(CH_2)_2C$ OOC_2H_5] (**13b**)*

Yield 84%; yellow oil; ^{1}H NMR: δ 7.25–7.27 (m, 4H), 7.09–7.23 (m, 5H), 5.32 (s, 1H), 4.00–4.08 (q, 2H, 7.2 Hz), 2.45–2.56 (m, 4H), 1.15–1.20 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 171.70, 141.11, 140.01, 133.01, 130.11, 128.79, 128.74, 127.37, 60.55, 47.19, 35.14, 19.72, 14.42; ^{77}Se NMR: δ 230.12; IR ($CHCl_3$, ν cm^{-1}): 1739.7.

*3.3.18. Ethyl 2-[(4-chlorophenyl)(phenyl)]methylselenobutanoate [(4-ClC_6H_4)(C_6H_5)$CHSe(CH_2)_3C$ OOC_2H_5] (**13c**)*

Yield 82%; yellow oil; ^{1}H NMR: δ 7.27–7.31 (m, 4H), 7.12–7.23 (m, 5H), 5.26 (s, 1H), 3.98–4.05 (q, 2H, 7.2 Hz), 2.32–2.38 (t, 2H, 7.2 Hz), 2.23–2.28 (t, 2H, 7.2 Hz), 1.79–1.84 (m, 2H), 1.15–1.19 (t, 3H, 7.2 Hz); ^{13}C NMR: δ 172.46, 141.12, 140.25, 132.94, 130.10, 128.75, 128.70, 127.30, 60.26, 46.94, 34.15, 25.21, 25.11, 14.43; ^{77}Se NMR: δ 228.50; IR ($CHCl_3$, ν cm^{-1}): 1732.5.

*3.4. Synthesis of pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids **20–25**(**a–c**)*

The pyridyl/naphthyl/(diphenyl)methylseleno substituted alkanoic acids **20–25**(**a–c**) were synthesized by the procedure as reported earlier for the preparation of phenyl/benzylseleno substituted alkanoic acids [40] in the cited reference.

*3.4.1. 2-Pyridylselenoethanoic acid [(C_5H_4N)$SeCH_2COOH$] (**20a**)*

Yield 90%; colorless crystalline solid; m.p. 120–122 °C; ^{1}H NMR: δ 10.53 (br s,1H), 8.45–8.47 (d, 1H, 5.1 Hz), 7.64–7.68 (t, 1H, 7.6 Hz), 7.54–7.56 (d, 1H, 8.0 Hz), 7.26–7.29 (t, 1H, 5.1 Hz), 3.67 (s, 2H); ^{13}C NMR: δ 171.75, 148.98, 138.0, 126.54, 122.14, 26.78; ^{77}Se NMR: δ 391.28; IR (KBr, ν cm^{-1}): 1718.2; MS-EI, *m/e* (R.I., assignment): 218 (100, $[M+2]^+$), 216 (46, $[M]^+$). Anal. Calc. for $C_7H_7NO_2Se$: C, 38.90; H, 3.26; N, 6.48. Found: C, 38.82; H, 3.27; N, 6.35%.

*3.4.2. 2-Pyridylselenopropanoic acid [(C_5H_4N)$Se(CH_2)_2COOH$] (**20b**)*

Yield 88%; colorless crystalline solid; m.p. 60–61 °C; ^{1}H NMR: δ 9.87 (br s,1H), 8.29–8.31 (d, 1H, 4.8 Hz), 7.24–7.29 (t, 1H, 7.5 Hz), 7.11–7.16 (d, 1H, 4.8 Hz), 6.77–6.82 (t, 1H, 7.2 Hz), 3.26–3.32 (t, 2H, 7.12 Hz); ^{13}C NMR: δ 171.7, 155.14, 150.02, 135.47, 125.29, 119.90, 35.47, 19.38; ^{77}Se NMR: δ 387.70; IR (KBr, ν cm^{-1}): 1715.6. Anal. Calc. for $C_8H_9NO_2Se$: C, 41.75; H, 3.94; N, 6.08. Found C, 41.70; H, 3.92; N, 6.14%.

*3.4.3. 2-Pyridylselenobutanoic acid [(C_5H_4N)$Se(CH_2)_3COOH$] (**20c**)*

Yield 81%; colorless crystalline solid; m.p. 40–42 °C; ^{1}H NMR: δ 8.44 (br s,1H), 7.17–7.74 (d, 1H, 5.6 Hz), 7.52–7.55 (d, 1H, 5.7 Hz), 7.46–7.49 (t, 1H, 6.6 Hz), 7.05–7.08 (t, 1H, 5.6 Hz), 3.22–3.26 (t, 2H, 7.0 Hz), 2.52–2.56 (t, 2H, 7.0 Hz), 2.07–2.20 (m, 2H); ^{13}C NMR: δ 177.70, 167.81, 149.80, 136.46, 131.01, 128.91, 125.83, 120.65, 33.82, 27.77, 25.74, 19.22; ^{77}Se NMR: δ 381.21; IR (KBr, ν cm^{-1}): 1721.3. Anal. Calc. for $C_9H_{11}NO_2Se$: C, 44.26; H, 4.54; N, 5.76. Found: C, 44.27; H, 4.63; N, 5.45%.

*3.4.4. 2-(5-Methyl)pyridylselenoethanoic acid [(5-$CH_3C_5H_3N$)$SeCH_2$-COOH] (**21a**)*

Yield 92%; yellowish-white solid; m.p. 57–59 °C; ^{1}H NMR: δ 10.78 (br s,1H), 8.23 (s, 1H), 7.33–7.42 (m, 2H), 3.51 (s, 2H), 2.22 (s, 3H); ^{13}C NMR: δ 171.33, 151.10, 138.72, 131.91, 125.82, 29.76, 26.59, 17.91; ^{77}Se NMR: δ 371.4; IR (KBr, ν cm^{-1}): 1728.7. Anal. Calc. for $C_8H_9NO_2Se$: C, 41.75; H, 3.94; N, 6.09. Found C, 43.56; H, 4.48; N, 6.15%.

*3.4.5. 2-(5-Methyl)pyridylselenopropanoic acid [(5-$CH_3C_5H_3N$)Se-$(CH_2)_2COOH$] (**21b**)*

Yield 91%; yellowish-white solid; m.p. 49–51 °C; ^{1}H NMR: δ 10.62 (br s, 1H), 8.22 (s, 1H), 7.31–7.39 (m, 2H), 3.18–3.23 (t, 2H, 7.2 Hz), 2.36–2.41 (t, 2H, 7.2 Hz), 2.20 (s, 3H); ^{13}C NMR: δ 174.71, 151.01, 150.01, 138.25, 131.11, 126.10, 34.62, 24.12, 17.55; ^{77}Se NMR: δ 353.21; IR ($CHCl_3$, ν cm^{-1}): 1731.4. Anal. Calc. for $C_9H_{11}NO_2Se$: C, 44.27; H, 4.54; N, 5.74. Found: C, 44.29; H, 4.59; N, 5.61%.

*3.4.6. 2-(5-Methyl)pyridylselenobutanoic acid [(5-$CH_3C_5H_3N$)$Se(CH_2)_3$-COOH] (**21c**)*

Yield 89%; yellow oil; ^{1}H NMR: δ 10.11 (br s, 1H), 8.21 (s, 1H), 7.15–7.26 (m, 2H), 3.10–3.15 (t, 2H, 7.2 Hz), 2.39–2.44 (m, 2H), 2.20 (s, 3H), 1.90–2.03 (t, 2H, 6.9 Hz); ^{13}C NMR: δ 178.22, 150.95, 150.21, 137.13, 130.0, 125.45, 33.85, 25.71, 24.92, 17.95; ^{77}Se NMR: δ 345.11; IR ($CHCl_3$, ν cm^{-1}): 1708.9.

*3.4.7. 2-(5-Bromo)pyridylselenoethanoic acid [(5-BrC_5H_3N)$SeCH_2COOH$] (**22a**)*

Yield 76%; yellowish-white solid; m.p. 96–97 °C; ^{1}H NMR: δ 10.02 (br s, 1H), 8.48–8.49 (d, 1H, 2.1 Hz), 7.62–7.66 (d, 1H, 8.4 Hz), 7.30–7.33 (d, 1H, 8.4 Hz), 3.65 (s, 2H); ^{13}C NMR: δ 171.68, 153.35, 150.62, 139.96, 126.90, 118.57, 26.35; ^{77}Se NMR: δ 380.04; IR (KBr, ν cm^{-1}): 1718.2. Anal. Calc. for $C_7H_6BrNO_2Se$: C, 28.40; H, 2.38; N, 4.75. Found: C, 30.95; H, 2.46; N, 4.67%.

*3.4.8. 2-(5-Bromo)pyridylselenopropanoic acid [(5-BrC_5H_3N)$Se(CH_2)_2$-COOH] (**22b**)*

Yield 74%; yellowish-white solid; m.p. 92–93 °C; ^{1}H NMR: δ 9.96 (br s, 1H), 8.32–8.34 (d, 1H, 2.1 Hz), 7.60–7.64 (d, 1H, 8.4 Hz), 7.24–7.27 (d, 1H, 8.4 Hz), 3.16–3.21 (t, 2H, 7.2 Hz), 2.40–2.45 (t, 2H, 7.2 Hz); ^{13}C NMR: δ 171.21, 152.89, 150.61, 139.55, 126.15, 118.02, 34.11, 26.02; ^{77}Se NMR: δ 376.09; IR ($CHCl_3$, ν cm^{-1}): 1716.1. Anal. Calc. for $C_8H_8BrNO_2Se$: C, 31.09; H, 2.61; N, 4.53. Found: C, 31.14; H, 2.65; N, 4.82%.

*3.4.9. 2-(5-Bromo)pyridylselenobutanoic acid [(5-BrC_5H_3N)$Se(CH_2)_3$-COOH] (**22c**)*

Yield 71%; yellowish-white semisolid; ^{1}H NMR: δ 9.85 (br s, 1H), 8.27–8.29 (d, 1H, 2.1 Hz), 7.55–7.59 (d, 1H, 8.4 Hz), 7.22–7.26 (d, 1H, 8.4 Hz), 3.08–3.13 (t, 2H, 7.2 Hz), 2.37–2.42 (m, 2H), 1.77–1.83 (t, 2H, 6.9 Hz); ^{13}C NMR: δ 170.91, 152.36, 149.69, 139.01, 125.92, 117.90, 34.01, 25.95, 19.12; ^{77}Se NMR: δ 369.21; IR ($CHCl_3$, ν cm^{-1}): 1714.2.

*3.4.10. 2-Naphthylselenoethanoic acid [(1-$C_{10}H_7$)$SeCH_2COOH$] (**23a**)*

Yield 89%; colorless crystalline solid; m.p. 39–40 °C; ^{1}H NMR: δ 9.13 (br s, 1H), 8.36–8.38 (d, 1H, 8.3 Hz), 7.88–7.90 (d, 1H, 6.9 Hz), 7.80–7.82 (d, 2H, 7.9 Hz), 7.47–7.56 (m, 2H), 7.33–7.37 (t, 1H, 7.9 Hz), 3.48 (s, 2H); ^{13}C NMR: δ 177.10, 134.24, 134.11, 133.99, 129.74, 128.78, 127.93, 127.35, 127.07, 126.37, 125.85, 27.21; ^{77}Se NMR: δ 275.14; IR (KBr, ν cm^{-1}): 1705.2; MS-EI, *m/e* (R.I., assignment): 283 (100, $[M+18]^+$), 265 (14, $[M]^+$). Anal. Calc. for $C_{12}H_{10}O_2Se$: C, 54.35; H, 3.80. Found: C, 54.37; H, 3.91%.

*3.4.11. 2-Naphthylselenopropanoic acid [(1-$C_{10}H_7$)$Se(CH_2)_2COOH$] (**23b**)*

Yield 83%; colorless crystalline solid; m.p. 41–42 °C; ^{1}H NMR: δ 8.98 (br s, 1H), 8.34–8.36 (d, 1H, 8.4 Hz), 7.81–7.83 (d, 1H, 7.6 Hz), 7.78–7.80 (d, 1H, 7.6 Hz), 7.46–7.57 (m, 2H), 7.33–7.37 (t, 2H, 7.6 Hz), 3.06–3.08 (t, 2H, 7.4 Hz), 2.63–2.67 (t, 2H, 7.4 Hz); ^{13}C NMR: δ 178.12, 133.91, 133.87, 133.30, 128.89, 128.65, 128.22, 127.32, 126.81, 126.32, 125.71, 35.42, 22.03; ^{77}Se NMR: δ 254.41; IR (KBr, ν cm^{-1}): 1708.4. Anal. Calc. for $C_{13}H_{12}O_2Se$: C, 55.93; H, 4.33. Found: C, 56.04; H, 4.39%.

*3.4.12. 2-Naphthylselenobutanoic acid [(1-$C_{10}H_7$)$Se(CH_2)_3COOH$] (**23c**)*

Yield 77%; colorless crystalline solid; m.p. 43–44 °C; ^{1}H NMR: δ 8.71 (br s, 1H), 8.35–8.37 (d, 1H, 8.2 Hz), 7.79–7.81 (d, 1H, 7.6 Hz), 7.74–7.76 (d, 2H, 7.6 Hz), 7.45–7.54 (m, 2H), 7.32–7.35 (t, 1H, 7.6 Hz), 2.92–2.96 (t, 2H, 7.2 Hz), 2.43–2.46 (t, 2H, 7.1 Hz), 1.90–1.97 (m, 2H); ^{13}C NMR: δ 179.12, 134.32, 134.02, 132.38, 129.02, 128.69, 128.40, 127.54, 126.78, 126.25, 125.80, 34.06, 27.13, 25.11; ^{77}Se NMR: δ 248.19; IR (KBr, ν cm^{-1}): 1710.1. Anal. Calc. for $C_{14}H_{14}O_2Se$: C, 57.35; H, 4.81. Found: C, 57.29; H, 4.75%.

*3.4.13. 2-(Diphenyl)methylselenoethanoic acid [(C_6H_5)$_2CHSeCH_2$-COOH] (**24a**)*

Yield 87%; colorless crystalline solid; m.p. 96–99 °C; ^{1}H NMR: δ 9.70 (br s, 1H), 7.38–7.41 (d, 4H, 7.5 Hz), 7.11–7.26 (m, 6H), 5.64 (s, 1H), 2.88 (s, 2H); ^{13}C NMR: δ 177.93, 140.37, 129.02, 128.73, 127.51, 48.93, 24.03; ^{77}Se NMR: δ 247.16; IR (KBr, ν cm^{-1}): 1715.1. Anal. Calc. for $C_{15}H_{14}O_2Se$: C, 59.02; H, 4.62. Found: C, 58.93; H, 4.15%.

*3.4.14. 2-(Diphenyl)methylselenopropanoic acid [$(C_6H_5)_2CHSe(CH_2)_2$-COOH] (**24b**)*

Yield 86%; white solid; m.p. 88–89 °C; ^{1}H NMR: δ 9.65 (br s, 1H), 7.32–7.42 (m, 4H), 7.05–7.19 (m, 6H), 5.30 (s, 1H), 2.35–2.44 (m, 4H); ^{13}C NMR: δ 175.95, 140.10, 130.91, 129.74, 127.55, 48.54, 35.24, 19.75, 14.48; ^{77}Se NMR: δ 241.11; IR ($CHCl_3$, ν cm^{-1}): 1721.3. Anal. Calc. for $C_{16}H_{16}O_2Se$: C, 60.19; H, 5.05. Found C, 59.87; H, 4.81%.

*3.4.15. 2-(Diphenyl)methylselenobutanoic acid [$(C_6H_5)_2CHSe(CH_2)_3$-COOH] (**24c**)*

Yield 81%; white semisolid; ^{1}H NMR: δ 9.62 (br s, 1H), 7.30–7.44 (m, 4H), 7.01–7.13 (m, 6H), 5.26 (s, 1H), 2.28–2.33 (t, 2H, 7.2 Hz), 2.17–2.21 (t, 2H, 7.2 Hz), 1.72–1.77 (m, 2H); ^{13}C NMR: δ 175.64, 140.03, 130.75, 129.18, 127.44, 48.32, 35.19, 22.36, 19.75, 14.48; ^{77}Se NMR: δ 237.91; IR ($CHCl_3$, ν cm^{-1}): 1741.2.

*3.4.16. 2-[(4-Chlorophenyl)(phenyl)]methylselenoethanoic acid [$(4\text{-}ClC_6H_4)(C_6H_5)CHSeCH_2COOH$] (**25a**)*

Yield 85%; colorless crystalline solid; m.p. 85–87 °C; ^{1}H NMR: δ 10.08 (br s, 1H), 7.32–7.36 (m, 4H), 7.09–7.28 (m, 5H), 5.51 (s, 1H), 2.80 (s, 2H); ^{13}C NMR: δ 170.65, 140.11, 139.15, 133.11, 130.18, 128.26, 128.11, 127.52, 47.55, 23.92; ^{77}Se NMR: δ 241.39; IR (KBr, ν cm^{-1}): 1714.4. Anal. Calc. for $C_{17}H_{17}ClO_2Se$: C, 55.03; H, 3.85. Found: C, 55.17; H, 3.96%.

*3.4.17. 2-[(4-Chlorophenyl)(phenyl)]methylselenopropanoicacid [$(4\text{-}ClC_6H_4)(C_6H_5)CHSe(CH_2)_2CO\ OH$] (**25b**)*

Yield 79%; yellow solid; m.p. 79–80 °C; ^{1}H NMR: δ 10.01 (br s, 1H), 7.23–7.29 (m, 4H), 7.07–7.25 (m, 5H), 5.44 (s, 1H), 2.40–2.46 (m, 4H); ^{13}C NMR: δ 171.45, 140.98, 140.85, 132.77, 130.09, 128.76, 128.69, 127.32, 47.09, 34.08, 19.52; IR ($CHCl_3$, ν cm^{-1}): 1723.7. Anal. Calc. for $C_{18}H_{19}ClO_2Se$: C, 56.78; H, 4.76. Found C, 56.71; H, 4.84%.

*3.4.18. 2-[(4-Chlorophenyl)(phenyl)]methylselenobutanoicacid [$(4\text{-}ClC_6H_4)(C_6H_5)CHSe(CH_2)_3COO\ H$] (**25c**)*

Yield 75%; yellow semisolid; ^{1}H NMR: δ 9.89 (br s, 1H), 7.19–7.24 (m, 4H), 7.05–7.21 (m, 5H), 5.35 (s, 1H), 2.30–2.36 (t, 2H, 7.2 Hz), 2.21–2.27 (t, 2H, 7.2 Hz), 1.67–1.72 (m, 2H); ^{13}C NMR: δ 172.08, 141.07, 140.90, 132.88, 130.08, 128.66, 128.58, 127.22, 46.52, 34.18, 25.02, 24.92; IR ($CHCl_3$, ν cm^{-1}): 1742.9.

Acknowledgements

We gratefully acknowledge the financial support for this work from Department of Science and Technology (DST), New Delhi, Government of India, Project No. SR/FTP/CS-135/2006 dated 13-03-2007 and Council of Scientific and Industrial Research (CSIR), New Delhi, vide sanction Nos. F.NO.10-2(5)/2004(ii)-E.U.II dated 28-08-2004 and F.NO.10-2(5)/2005(i)-E.U.II dated 01-12-2005.

We are also thankful to Prof. Vitor Felix, Department of Chemistry and CICECO, University of Aveiro, 3810-193 Aveiro (Portugal), Mr. Kamlesh, Indian Institute of Technology, Kanpur (India) and Mr. Pallepogu Raghavaiah, School of Chemistry, University of Hyderabad, Hyderabad (India) for X-ray diffraction studies of the crystals of the compounds **20a**, **23a** and **24a**, respectively. Further, we wish to express our sincere thanks to Mr. Kamlesh for his co-operation at needful time in providing X-ray structures of compounds **20a**, **23a** and **24a** showing hydrogen-bonding interactions.

Appendix A. Supplementary material

CCDC 689426, 693852 and 689427 contain the supplementary crystallographic data for **20a**, **23a** and **24a**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2008.10.020.

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