First Isolation and Characterization of Sodium and Potassium Tellurocarboxylates: Structural Analysis of *Te*-Alkyl Telluroester

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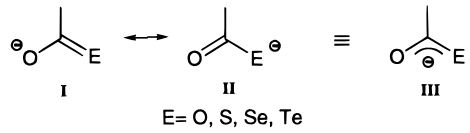
The first synthesis and characterization of sodium and potassium tellurocarboxylates were achieved by the reaction of acyl chlorides with the corresponding alkali metal tellurides. The salts are yellow to red solids or oils. The aliphatic derivatives are very sensitive toward oxygen and very thermally sensitive. However, the aromatic derivatives are relatively stable and can be stored for 1 week below −5 °C and under oxygen-free conditions. The most practical method for the synthesis of *Te*-alkyl telluroesters was established by the reaction of the sodium and potassium tellurocarboxylates with alkyl iodides at 0 °C. The first X-ray structural analysis of telluroesters (RCOTeR′) was carried out for *Te*-methyl 4-chlorobenzenecarbotelluroate (4-$ClC_6H_4COTeCH_3$), whose crystals are monoclinic ($P2_1/a$) with $a = 5.975(3)$ Å, $b = 14.517(2)$ Å, $c = 10.617(3)$ Å, $\beta = 92.74(3)°$, $V = 919.8(4)$ Å^3, and $Z = 4$. The molecule was nearly planar. The C═O and C−Te bond lengths are 1.204(3) and 2.153(3) Å, respectively, indicating C═O double and C−Te single bonds. The C−Te−C angle of the $C(O)TeCH_3$ moiety is close to a right angle (92.3°), much more narrow compared with those [C−E−C, E = O (>105°), S (>102°), Se (>95)] of common esters and thio- and selenoesters (ArC(O)ER′, E = O, S, Se; R′ = alkyl). The ν(C═O) bands and the ^{13}C═O and ^{125}Te NMR spectra of the sodium and potassium tellurocarboxylates are discussed in comparison with those of other alkali metal or oxygen, sulfur, and selenium isologues.

Introduction

As mentioned in the preceding paper, there are 15 possible kinds of alkali metal chalcogenocarboxylates for each alkali metal in which one or two O atoms of the carboxyl group were replaced by S, Se, or Te.[1] Although these alkali metal salts are the most important class of compounds for the synthesis of chalcogenocarboxylic acid derivatives and are considered to belong to the *heteroallylic anion* system **I**−**III** (Scheme 1), much less is known about the chemistry of these chalcogenocarboxylic acid salts except for the thio and dithio salts.[2] In particular, the synthesis of the tellurium isologues has remained largely unexplored probably due to their extreme instability.[3] We have reported the synthesis of lithium tellurocarboxylates by reaction of acyl chlorides with lithium telluride.[4] In addition, sodium tellurocarboxylates were found to be formed by reaction of acyl chlorides with sodium tellurides.[5] However, most of these salts were oils which contained the corresponding metal chloride and/or the solvents. An alternative method for reaction of diacyl tellurides with alkoxides where undesirable metal chlorides are not formed was also investigated, although it was limited to potassium 2-methoxybenzenecarbotelluroate[6a] and sodium pentanecarbotelluroate.[6b] The development of general synthetic method of sodium and potassium tellurocarboxylates **IV** has been required (Chart 1).

Scheme 1



Carboxylic acid and thiocarboxylic acid esters have been extensively investigated.[7] They are industrially produced and widely employed. In contrast, little is known about the chemistry of seleno- and telluroesters **V** due to the difficulty of their synthesis. To our knowlegde, no *Te*-alkyl telluroesters had been reported when our study began in 1968, most likely due to the difficulty of the synthesis of the starting compounds such as alkali metal tellurocarboxylates and alkanetellurolates.[8] The first synthesis of *Te*-alkyl telluroester was reported in 1970 by Piette and Renson, who prepared *Te*-butyl telluroaroylates by reacting

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(2) For reviews on thio-, dithio-, and selenocarboxylic acid derivatives: Kato, S.; Murai, T. In *Supplement B: The Chemistry of Acid Derivatives Vol. 2*; Patai, S., Eds.; John Wiley & Sons: New York, 1992; pp 803−847. Kato S.; Ishida, M. *Sulfur Rep.* **1988**, *8*, 155. Scheithauer, S.; Mayer, R. In *Topics in Sulfur Chemistry*; Senning, A., Eds.; Georg Thieme Publishers: Stuttgart, 1979; Vol. 4.
(3) For reviews on alkali metal tellurocarboxylates: (a) Ogawa A.; Sonoda, N. In *Comprehensive Organic Synthesis*; Moody, C. J., Ed.; Pergamon Press: Oxford, 1995; Vol. 5, pp 231−255. (b) Ishii, A.; Nakayama, J. in *Comprehensive Organic Synthesis*; Moody, C. J., Ed.; Pergamon Press: Oxford, 1995; Vol. 5, pp 505−543. (c) Kato, S.; Murai, T.; Ishida, M. *Org. Prep. Proced. Int.* **1986**, *18*, 369.
(4) Kato, S.; Sasaki, H.; Yagihara, M. *Phosphorus, Sulfur Silicon Relat. Elem.* **1992**, *67*, 27.
(5) Kanda, T.; Nakaiida, S.; Murai, T.; Kato, S. *Tetrahedron Lett.* **1989**, 1829.
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(7) (a) *The Chemistry of Carboxylic Acids and Esters*; Patai, S., Ed.; Wiley-Interscience: New York, 1969. (b) *The Chemistry of Acid Derivatives, Supplement B, Vol. 2, Parts 1 and 2*; Patai, S., Ed.; Wiley-Interscience: New York, 1992. (c) Mulzer, J. In *Comprehensive Organic Functional Group Transformations;* Moody, C. J., Ed.; Pergamon Press: Oxford, 1995; Vol. 5, pp 121−180.
(8) Alkali metal alkanetellurolates except for lithium butanetellurolate are very difficult to prepare.

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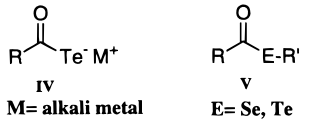
Chart 1

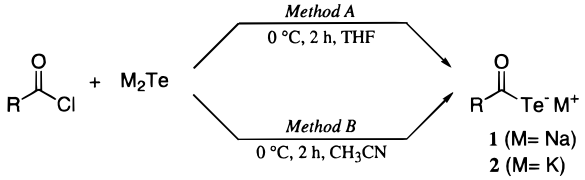


aroyl chlorides with lithium butanetellurolate.[9] Later, two synthetic methods were reported, including treating aroyl chlorides and/or alkyl iodides with sodium telluride in water[10] or sodium hydrogen telluride generated by reacting tellurium with sodium borohydride.[11] Recently selenoesters and *Te*-alkyl tellurobenzoate have been reported to be effective as liquid crystals,[12] as blocking agent of a number of different nerve impulses,[13] and as an acyl anion precursor.[14] We report here a general synthetic method of solvent-free and metal chloride free sodium tellurocarboxylates **1** and potassium tellurocarboxylates **2** and the synthesis and characterization of a series of *Te*-alkyl telluroesters **3** together with the first X-ray structural analysis of a telluroester.

Results and Discussion

Sodium and Potassium Tellurocarboxylates. The conditions for the synthesis of salts **1** and **2** were examined in detail by using 4-methylbenzoyl chloride and sodium or potassium telluride. The reactions were found to proceed readily at 0 °C in tetrahydrofuran (THF) to give sodium (**1n**) and potassium 4-methylbenzenecarbotelluroates (**2h**) in yields of 90% and 79%, respectively (method A in Scheme 2). Under the same conditions, other sodium (**1a**−**g**,**i**,**k**−**m**,**o**−**q**,**s**−**u**) (Table 1) and potassium tellurocarboxylates (**2a**,**c**−**g**,**j**,**k**) (Table 2) were synthesized in yields of 70−95%. The obtained salts, however, were oils which contained a small amount of the solvent, which was difficult to remove even under reduced pressure for over 20 h. After several disappointing attempts, crystalline **1** and **2** were obtained by using acetonitrile instead of THF (method B in Scheme 2). For example, when an acetonitrile solution of benzoyl chloride was slowly added to a suspension of excess sodium telluride in the same solvent at 0 °C, the solution quickly changed to dark red. After stirring at the same temperature for 2 h, the insoluble part (NaCl and a trace of black tellurium) was filtered out. Evaporation of the solvent under reduced pressure, crystallization, and then recrystallization of the resulting residue from a mixture of acetonitrile/ether/hexane gave sodium benzenecarbotelluroate (**1k**) as yellow plates in a yield of 79%. Similarly, the reactions with other acyl chlorides led to sodium tellurocarboxylates (**1a**−**f**,**h**−**j**,**l**,**n**−**t**,**v**) in yields of

Scheme 2



No.	R	No.	R
1a, **2a**	CH_3	**1m**	3-$CH_3C_6H_4$
1b	C_2H_5	**1n**, **2h**	4-$CH_3C_6H_4$
1c, **2b**	C_3H_7	**1o**, **2i**	2-$CH_3OC_6H_4$
1d, **2c**	*i*-C_3H_7	**1p**, **2j**	4-$CH_3OC_6H_4$
1e	C_4H_9	**1q**	2-ClC_6H_4
1f, **2d**	*t*-C_4H_9	**1r**	3-ClC_6H_4
1g, **2e**	C_5H_{11}	**1s**, **2k**	4-ClC_6H_4
1h	$C_{17}H_{35}$	**1t**	3-Cl,2,6-$(CH_3O)_2$-C_6H_2
1i	*cyclo*-C_6H_{11}		
1j	1-Adamantyl	**1u**	1-Naph
1k, **2f**	C_6H_5	**1v**	2-Naph
1l, **2g**	2-$CH_3C_6H_4$		

40−80%. Moreover, the reaction with potassium telluride instead of sodium telluride under the same conditions gave potassium tellurocarboxylates (**2a**−**d**,**f**−**k**) in analogous yields. Their structures were established by IR and ^{1}H, ^{13}C, and ^{125}Te NMR spectra, and by elemental analysis or by conversion into *Te*-alkyl telluroesters **3**.

The crystalline sodium salts **1** and potassium salts **2** were pale yellow to yellow for **1** and yellow to red crystals for **2**.[15] In contrast, the salts which include THF[16] are red to dark red oils.[17] They are oxygeno- and thermo-labile. In particular, the aliphatic salts are extremely sensitive toward oxygen. For example, upon exposure to air the methyl derivatives **1a**,**b** and ethyl derivatives **2a** immediately liberate black tellurium and completely decompose within 1 min. Under oxygen-free conditions at temperatures below −20 °C, however, the aromatic derivatives are relatively stable and do not show appreciable changes in air for 30 min, and under oxygen-free conditions they can be stored at below −5 °C for at least 1 week. **1** and **2** are less soluble in nonpolar solvents such as benzene, ether,[18] and dichloromethane but, except for sodium 2-methyl- (**1l**) and 2-chlorotellurobenzoates **(1q**), are soluble in polar solvents such as methanol, tetrahydrofuran, and acetonitrile.

In Table 3 the carbonyl stretching frequencies and ^{13}C═O and ^{125}Te NMR chemical shifts are shown. The carbonyl stretching frequencies of the aliphatic salts are at a higher wavenumber than those of the aromatic counterparts (RCOTeNa, 1574−1584 cm^{-1} for R = alkyl and 1531−1567 cm^{-1} for R = aryl; RCOTeK, 1556−1588 cm^{-1} for R = alkyl and 1519−1548 cm^{-1} for R = aryl). In addition, the bands of the potassium salts **2**, except for **2a** and **2g**, appear at a lower frequency than those of the corresponding sodium salts **1**. The carbonyl carbon chemical shifts occur in a relatively narrow region of δ 206−

(9) (a) Piette, J. L.; Renson, M. *Bull. Soc. Chim. Belg.* **1970**, *79*, 383. (b) Piette, J. L.; Debergh, D.; Baiwir, M.; Llabres, L. *Spectrochim. Acta* **1980**, *36A*, 733.

(10) The preparation of four *Te*-benzyl telluroesters from the reaction of RCOCl and R′I with Na_2Te generated by treating Te with $NaBH_4$: Bergman, J.; Engman, L. *Z. Naturforsch.* **1980**, *35b*, 217.

(11) Four *Te*-alkyl esters (PhCOTeEt, PhCOTePr, PhCOTePr-*i*, and *n*-C_7H_{15}COTeEt) were prepared by the reaction of RCOCl with NaHTe which was generated by treating tellurium with sodium borohydride in ethanol, followed by alkyl iodides: Suzuki, H.; Inamoto, T.; Ogawa, T.; Tani, H. *J. Chem. Res., Synop.* **1990**, 56.

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(15) In general, *o*-methyl-substituted alkali metal chalcogenocarboxylates are less crystallizable than other derivatives.

(16) Attempts to remove tetrahydrofuran in the oily salts was unsuccessful even under various conditions.

(17) When the salts were dissolved in polar solvents such as methanol and THF, their color immediately changed to red to dark red. However, the absorption maximum was not observed in the visible region.

(18) 4-Methyl (**1n**, **2h**) and 4-methoxy derivatives (**1p**, **2j**) are less soluble in ether than the aliphatic (**1a**−**j**, **2a**−**e**), benzene (**1k**, **2f**) and 2-methylbenzene derivatives (**1l**, **2g**).

Table 1. Yields of Sodium Tellurocarboxylates **1**, $RCOTe^-Na^+$

no.	R	yield[a] (%) [meth]	note[b]	no.	R	yield[a] (%) [meth]	note[b]
1a	CH_3	85 [A]	DR, oil	**1l**	2-$CH_3C_6H_4$	96 [A]	DR, oil
		36 [B]	PY, cryst			96 [B]	DR, oil
1b	C_2H_5	90 [A]	DR, oil	**1m**	3-$CH_3C_6H_4$	82 [A]	DR, oil
		59 [B]	PY, cryst	**1n**	4-$CH_3C_6H_4$	90 [A]	DR, oil
1c	C_3H_7	92 [A]	DR, oil			78 [B]	Y, cryst
		56 [B]	PY, cryst	**1o**	2-$CH_3OC_6H_4$	79 [A]	DR, oil
1d	*i*-C_3H_7	78 [A]	DR, oil			39 [B]	RO, cryst
		50 [B]	PY, cryst	**1p**	4-$CH_3OC_6H_4$	85 [A]	DR, oil
1e	C_4H_9	85 [A]	DR, oil			62 [B]	Y, cryst
		45 [B]	PY, cryst	**1q**	2-ClC_6H_4	92 [A]	DR, oil
1f	*t*-C_4H_9	80 [A]	DR, oil			88 [B]	RO, oil
		63 [B]	PY, cryst	**1r**	3-ClC_6H_4	62 [B]	Y, cryst
1g	C_5H_{11}	85 [A]	DR, oil	**1s**	4-ClC_6H_4	80 [A]	DR, oil
		82 [C]	O, cryst			59 [B]	Y, cryst
1h	$C_{17}H_{35}$	59 [B]	W, cryst	**1t**	3-Cl,2,6-$(CHO_3)_2C_6H_2$	92 [A]	DR, oil
1i	c-C_6H_{11}	75 [A]	DR, oil			77 [B]	Y, cryst
		68 [B]	PY, cryst	**1u**	1-Naph	78 [A]	RO, oil
1j	1-adamantyl	79 [B]	PY, cryst	**1v**	2-Naph	76 [B]	O, cryst
1k	C_6H_5	80 [A]	DR, oil				
		79 [B]	Y, cryst				

[a] Method A: RCOCl + Na_2Te in tetrahydrofuran. Method B: RCOCl + Na_2Te in acetonitrile. Method C: $(RCO)_2Te$ + EtONa in ether. The yields of method A are the converted yield into *Te*-methyl or *Te*-ethyl telluroesters. [b] DR = dark red. PY = pale yellow. O = orange. RO = reddish orange. Y = yellow. W = white.

Table 2. Yields of Potassium Tellurocarboxylates **2**, $RCOTe^-K^+$

no.	R	yield[a] (%) [meth]	note[b]	no.	R	yield[a] (%) [meth]	note[b]
2a	CH_3	87 [A]	Y, oil	**2g**	2-$CH_3C_6H_4$	90 [A]	DR, oil
		33 [B]	PY, cryst			66 [B]	O, cryst
2b	C_3H_7	66 [B]	PY, cryst	**2h**	4-$CH_3C_6H_4$	79 [A]	RO, oil
2c	*i*-C_3H_7	78 [A]	Y, oil			56 [B]	R, cryst
		45 [B]	PY, cryst	**2i**	2-$CH_3OC_6H_4$	61 [B]	DR, cryst
2d	*t*-C_4H_9	78 [A]	Y, oil	**2j**	4-$CH_3OC_6H_4$	85 [A]	DR, oil
		55 [B]	PY, cryst			62 [B]	O, cryst
2e	C_5H_{11}	82 [A]	O, oil	**2k**	4-ClC_6H_4	80 [A]	DR, oil
2f	C_6H_5	95 [A]	DR, oil			59 [B]	R, cryst
		55 [B]	R, cryst				

[a] Method A: RCOCl + K_2Te in tetrahydrofuran. Method B: RCOCl + K_2Te in acetonitrile. The yield method A is the converted yield into *Te*-methyl or *Te*-ethyl telluroesters. [b] DR = dark red; O = orange; R = red; RO = reddish orange; PY = pale yellow;Y = yellow. W = white.

Table 3. The νC=O Bands and ^{13}C=O and ^{125}Te NMR Spectral Data for Selected Sodium Tellurocarboxylates **1** and Potassium Tellurocarboxylates **2**

	RCOTeNa (**1**)				RCOTeNa (**2**)			
R	no.	νC=O (cm^{-1})[a]	^{13}C=O (δ)[b]	^{125}Te (δ)[c]	no.	νC=O (cm^{-1})[a]	^{13}C=O (δ)[b]	^{125}Te (δ)[c]
CH_3	**1a**	1584	207.8	251.9	**2a**	1588	207.8	250.4
C_2H_5	**1b**	1578	213.9	196.1				
i-C_3H_7	**1d**	1574	219.7	159.9	**2c**	1556	219.6	153.5
t-C_4H_9	**1f**	1574	223.6	63.0	**2d**	1556	223.1	61.2
C_6H_5	**1k**	1552	208.8	224.8	**2f**	1548	208.9	220.0
2-$CH_3C_6H_4$	**1l**	1531	213.1	394.0	**2g**	1532	213.0	390.8
4-$CH_3C_6H_4$	**1n**	1567	208.2	197.8	**2h**	1538	208.0	197.3
2-$CH_3OC_6H_4$	**1o**	1532	208.8	424.4	**2i**	1519	208.6	413.1
4-$CH_3OC_6H_4$	**1p**	1566	206.1	174.9	**2j**	1546	206.1	167.6
2-ClC_6H_4	**1q**	1534	208.3	455.3				
4-ClC_6H_4	**1s**	1559	206.8	240.7	**2k**	1536	207.1	237.7
2-Naph	**1v**	1562	208.8	231.8				

[a] Nujol. [b] CD_3OD. [c] CD_3OD, standard: $(CH_3)_2Te$.

224. The ^{125}Te signals are observed in the range δ 63−455 for **1** and δ 61−413 for **2**. The signals of the *tert*-butyl derivatives **1f** and **2d** appear in the most upfield region. The introduction of ortho substituent derivatives resulted in substantial downfield shifts and in addition those of the potassium salts **2** show an upfield shift of 0.5−11 ppm compared with those of the corresponding sodium salts **1**. Table 4 shows a comparison of the carbonyl stretching frequencies and the carbonyl ^{13}C and the ^{125}Te NMR chemical shifts of alkali metal tellurobenzoates. A certain trend of the carbonyl stretching frequency is not observed for the change of metal cations. The effects of metal cations on the carbonyl carbon chemical shifts of alkali metal selenocarboxylates appear to be very little,[19] while the ^{125}Te NMR signals show a downfield shift in the order potassium, rubidium, and cesium salts. Table 5 shows a comparison of the carbonyl stretching frequencies and the carbonyl carbon chemical shifts of sodium benzoate and thio-, seleno-, and tellurobenzoates. The νC=O bands show high wavenumber shifts in the order thio-, seleno-, and telluroesters, while the carbonyl carbon

(19) In the cases of alkali metal thio- and selenocarboxylates, the effects of the alkali metal cations on the carbonyl carbon chemical shifts are very little.[1,23]

Table 4. Spectal Data for Alkali Metal Benzenecarbotelluroates, RCOTeM

R	M	IR (cm^{-1})[a] νC=O	NMR (δ)[b] ^{13}C=O	^{125}Te[c]	ref
C_6H_5	Li	1558	211.1[d]	234.8[d]	4
	Na	1552	208.8	224.8	this work
	K	1548	208.9	220.0	this work
	Rb	1543	208.7	228.8	20
	Cs	1555	208.6	254.4	20

[a] Nujol. [b] CD_3OD. [c] Standard: $(CH_3)_2Te$. [d] $CDCl_3$.

Table 5. Spectral Data for Sodium Benzenecarbochalcogenoates, RCOENa

R	E	IR (cm^{-1})[a] νC=O	NMR (δ)[b] ^{13}C=O	E	ref
C_6H_5	O	1556[c]	175.6		*f*
	S	1521[22]	214.6[21a]		21a, 22
	Se	1525[23]	215.9[23]	361.6 (^{77}Se)[d,23]	23,24
			216.1[24]		
	Te	1552	208.8	224.8 (^{125}Te)[e]	this work

[a] Nujol. [b] CD_3OD. [c] KBr. [d] Standard: $(CH_3)_2Se$. [e] Standard: $(CH_3)_2Te$. [f] See Experimental Section (Materials).

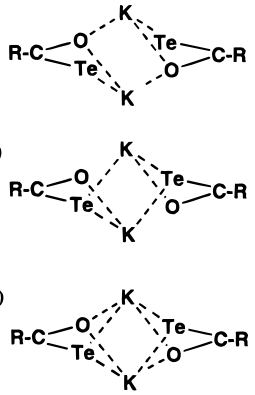


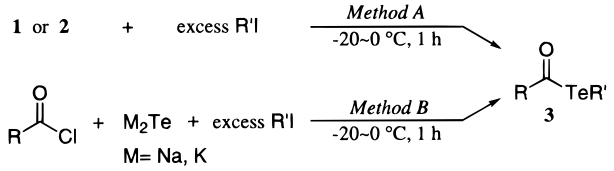
Figure 1. Structural modes of potassium arenecarbotelluroates.

signals do not show similar tendency. It is noted that the ^{13}C=O signal of the tellurobenzoate is observed at an upfield region compared to those of the thio- and selenobenzoates. Presumably such upfield shifts arised from the metallic property of tellurium.

It is well-known that the negative charge of carboxylic acid sodium salts is delocalized on the carboxyl group. The structural analysis of tellurocarboxylic acid sodium and potassium salts has not yet been reported. We have attempted to obtain single crystals of sodium and potassium tellurocarboxylates. Our efforts, however, have achieved unsuccessful results, even under various conditions. As shown in the preceding paper, we have revealed the structures of heavy alkali metal thiocarboxylates[1] and potassium[1] and cesium selenocarboxylates,[1,20b] and rubidium[1] and cesium tellurocarboxylates are dimeric.[1,20] On the basis of these structural analyses and the similarity of the IR and ^{13}C=O and ^{125}Te NMR spectra, the aromatic potassium salts (**2**, R = aryl) may be dimeric structures (**a**, **b**, or **c**, in Figure 1) in which the oxygen and/or tellurium atom is associated with the metal of the opposite molecule.

***Te*-Alkyl Telluroesters.** The successful preparation of sodium salts **1** and potassium salts **2** prompted us to synthesize and characterize of a series of *Te*-alkyl telluroesters **3**. In fact, salts **1** and **2** were found to readily react with alkylating agents such as alkyl iodides to give the expected telluroesters **3** in moderate to good yields,[25] but this required a large excess of alkyl iodides (method A in Scheme 3) (Table 6). To avoid decomposition of the salts, a one-pot reaction, consisting of the reaction of acyl chlorides with sodium telluride, followed by the addition of alkylating reagents, was performed (method B in Scheme 3). The yields of **3** increased to 70−90%.

Scheme 3



The resulting *Te*-alkyl esters **3** are yellow to reddish orange liquids or crystals. They are stable thermally, but labile toward oxygen. The aromatic esters (R = aryl) are more stable than the aliphatic esters. For example, *Te*-alkyl arenecarbotelluroates **3p**−**zq** show no appreciable changes for at least 10 min with exposure to air at room temperature, and they can be stored for 1 year under oxygen-free conditions in a refrigerator below −15 °C.

The carbonyl stretching bands and ^{13}C=O and ^{125}Te chemical shifts of the telluroesters **3** are shown in Table 7. The νC=O bands can be observed at 1650−1705 cm^{-1}, and the bands of the aliphatics appear at higher wavenumbers than those of the aromatic derivatives (RCOTeR′: 1680−1705 cm^{-1} for R = alkyl and 1655−1675 cm^{-1} for R = aryl), except for the *o*-methoxy derivatives, which show another peak at 1620−1630 cm^{-1}. The carbonyl carbon chemical shifts occur in the region δ 189−213, and the signals of the aliphatic derivatives (δ 197−213) (RCOTeR′: R = alkyl) appear upfield of those of the aromatic derivatives (δ 189−209) (RCOTeR′: R = aryl). Among the aliphatics, the carbonyl carbon signals show downfield shifts in the order R = CH_3, CH_2CH_3, $CH(CH_3)_2$, and $C(CH_3)_3$. A similar downfield shift is observed for the carbonyl carbon of *Se*-organyl selenoesters,[26] the selenocarbonyl carbon of *O*-silyl selenoesters,[27] and the thiocarbonyl carbon signals of dithioesters.[28] Among the aromatic derivatives, the *o*-methoxy substituent exhibits an upfield shift for the carbonyl carbon. The ^{125}Te signals can be observed in the range δ 540−831. The introduction of an *o*-methoxy group on the benzene ring produces pronounced downfield shifts of the Te nuclei in telluroesters.

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(21) Unpublished results: (a) C_6H_5COSNa: ^{13}C NMR (CD_3OD) δ 128.1, 129.1, 131.2, 145.5 (Ar), 214.6 (C=O). (b) $C_6H_5COSeNa$: ^{1}H NMR (CD_3OD) δ 7.27−7.41 (m, 3H, Ar), 8.10−8.12 (m, 2H, Ar); ^{13}C NMR (CD_3OD) δ 128.2, 129.3, 131.6, 147.9 (Ar), 215.9 (C=O); ^{77}Se NMR (CD_3OD) δ 362.6. (c) $C_6H_5COSCH_3$: IR (neat) 3062, 2929, 1664 (C=O), 1596, 1581, 1489, 1420, 1313, 1207, 1176, 1076, 1027, 1001, 968, 913, 773, 688, 648, 616 cm^{-1}; ^{1}H NMR (CD_3OD) δ 2.43 (s, 3H, SCH_3), 7.43 (t, J = 7.4 Hz, 2H, Ar), 7.55 (t, J = 7.4 Hz, 1H, Ar), 7.86 (d, J = 7.4 Hz, 2H, Ar); ^{13}C NMR (CD_3OD) δ 11.6 (CH_3), 127.1, 128.6, 133.2, 137.0 (Ar), 192.3 (C=O).

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(25) The use of a large excess of alkyl iodides led to short reaction times, resulting in high yields of the esters **3**. The reactions appear to proceed quantitatively. The low yields of the aliphatic telluroesters (**3a**, **3b**, **3f**, and **3h**) are considered to be due to the decomposition of the corresponding starting salts **1** and **2**.

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Table 6. Yields of *Te*-Alkyl Tellurocarboxylates **3**, RCOTeR′

no.	R	R′	yield[a] (%) A	yield[a] (%) B	bp (°C/Torr) or mp (°C)	note[b]
3a	CH_3	CH_3	86	63	42−48/30	OY, liq
3b		C_2H_5		39	58−60/30	O, liq
3c	C_2H_5	CH_3		56		O, liq
3d		C_2H_5		73		O, liq
3e	C_3H_7	CH_3	85	79	69−72/15	OY, liq
3f	*i*-C_3H_7	CH_3	77	62	57−59/16	OY, liq
3g	C_4H_9	CH_3	76	66	78−81/12	OY, liq
3h	*t*-C_4H_9	CH_3	85	69	80−82/70	O, liq
3i		C_2H_5		70		O, liq
3j	C_5H_{11}	CH_3		99	78−80/8	O, liq
3k		C_2H_5		83	92−94/8	O, liq
3l	$C_{17}H_{35}$	C_2H_5	59		30−31[c]	Y, cryst
3m	C_6H_{11}	CH_3	80	71	91−92/5	OY, liq
3n	1-adamantyl	CH_3	79	74		OY, liq
3o		*i*-C_4H_9	90		sublimable	PY, oil
3p	C_6H_5	CH_3	88	72	64−67/0.1	OY, liq
3q		C_2H_5		67	76−78/0.2	O, liq
3r		C_3H_7		80	82−84/0.1	O, liq
3s		$CH_2CH{=}CH_2$		71	78−80/0.1	O, liq
3t		$CH_2C{\equiv}CH$		68	76−80/0.1	O, liq
3u		CH_2CH_2OH		87		O, oil
3v	2-$CH_3C_6H_4$	CH_3	86	80	77−78/4	O, liq
					15−20[c]	Y, cryst
3w		C_2H_5	56			Y, liq
3x	3-$CH_3C_6H_4$	C_2H_5	53			O, liq
3y	4-$CH_3C_6H_4$	CH_3	95	71	76−78/0.1	O, liq
3z		C_2H_5		73	86−90/0.25	O, liq
3za	2-$CH_3OC_6H_4$	CH_3	85	74	101−103/0.1	O, liq
					20−25[c]	Y, cryst
3zb		C_2H_5		85	112−116/0.3	O, liq
3zc		C_3H_7		78	112−115/0.2	O, liq
3zd		*i*-C_3H_7	74			Y, liq
3ze		C_4H_9	70			Y, liq
3zf	4-$CH_3OC_6H_4$	CH_3	85	71	117−119/2.5	O, liq
3zg		C_2H_5	59			Y, liq
3zh	2-ClC_6H_4	C_2H_5	70			Y, liq
3zi	3-ClC_6H_4	C_2H_5	63			Y, liq
3zj	4-ClC_6H_4	CH_3	97	84	81−83[c]	Y, cryst
3zk		C_2H_5	68	66	90−93/0.1	O, liq
					25−30[c]	Y, cryst
3zl	3-Cl,2,6-$(CH_3O)_2C_6H_2$	CH_3	92			Y, oil
3zm		C_2H_5	86			Y, oil
3zn		*i*-C_4H_9	57			Y, oil
3zo	1-Naph	CH_3	77		26−28[c]	Y, cryst
3zp		C_2H_5	75			Y, oil
3zq	2-Naph	C_2H_5	62		61−63[c]	Y, cryst

[a] A = method A; B = method B. [b] O = orange; OR = orange red; OY = orange yellow; PY = pale yellow; Y = yellow. [c] Melting point.

Table 8 shows the carbonyl stretching frequencies and $^{13}C{=}O$ NMR spectra of methyl benzoate and its sulfur, selenium, and tellurium isologues. As in the sodium salts **1**, the $\nu C{=}O$ bands, except for methyl benzoate, show low wavenumber shifts in the order E = S, Se, and Te, while the carbonyl carbon signals of the telluroester show downfield shifts compared to those of the thio- and selenoesters.

It is well-known that common esters[32] and chalcogenoesters such as thiolesters,[33−36] thionesters,[35,37,38] and dithioesters[39] exist in the *Z*-configuration. Seleno- and telluroesters have not yet been subjected to a structural analysis. The stability of **3** allowed us to characterize it crystallographically. After several unsuccessful attempts to obtain single crystals of several *Te*-alkyl arenecarbotelluroates, we succeeded in obtaining single crystals of 4-chlorobenzenecarbotelluroate **3zj**. An ORTEP drawing of the molecular structure is shown in Figure 2. Final atomic

(28) ^{13}C NMR ($CDCl_3$): CH_3CSSCH_3, δ 231.3 (C=S); $C_2H_5CSSCH_3$, δ 239.8 (C=S); *i*-$C_3H_7CSSCH_3$, δ 247.0 (C=S); *t*-$C_4H_9CSSCH_3$, δ 254.2 (C=S).

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Table 7. Spectral Data for *Te*-Alkyl Tellurocarboxylates **3**

		NMR (δ)[b]		
no.	IR (cm−1)[a] νC═O	^{13}C═O	$^{13}CH_3Te$ or $^{13}CH_2Te$	^{125}Te[c]
3a	1702	196.8	−14.1 (CH_3Te)	663.5
3b	1705	197.0	3.6 (CH_2Te)	
3c	1700	202.3	−15.3 (CH_3Te)	
3d	1705	203.0	2.7 (CH_2Te)	
3e	1702	202.0	−14.8 (CH_3Te)	633.0
3f	1700	208.5	−15.3 (CH_3Te)	579.9
3g	1702	201.9	−14.9 (CH_3Te)	631.0
3h	1702	211.6	−15.5 (CH_3Te)	547.9
3i	1700	212.2	2.0 (CH_2Te)	
3j	1700	202.7	−15.0 (CH_3Te)	
3k	1700	202.1	2.7 (CH_2Te)	814.1
3l	1705	202.6	2.9 (CH_3Te)	
3m	1702	207.6	−15.4 (CH_3Te)	588.3
3n	1701	212.5	−16.2 (CH_3Te)	540.4
3o	1680	212.7	20.7 (CH_2Te)	
3p	1660	196.8	−14.0 (CH_3Te)	613.7
3q	1660	196.4	3.6 (CH_2Te)	793.6
3r	1655	196.1	14.0 (CH_2Te)	
3s	1655	195.9	13.5 (CH_2Te)	
3t	1655	209.0	−8.1 (CH_2Te)	
3u	1655	196.2	14.9 (CH_2Te)	
3v	1677	198.0	−12.8 (CH_3Te)	657.9
3w	1675	198.8	4.7 (CH_2Te)	
3x	1660	196.5	3.5 (CH_2Te)	
3y	1671	194.8	−14.2 (CH_3Te)	601.6
3z	1660	195.3	3.3 (CH_2Te)	781.8
3za	1630	189.3	−13.4 (CH_3Te)	761.2
3zb	1630	190.0	3.2 (CH_2Te)	
3zc	1630	189.7	13.3 (CH_2Te)	
3zd	1620	191.9	26.1 (CH)	
3ze	1623	189.8	10.6 (CH_2Te)	
3zf	1669	192.7	−14.4 (CH_3Te)	584.0
3zg	1665	193.5	3.3 (CH_2Te)	
3zh	1670	195.2	5.7 (CH_2Te)	
3zi	1670	195.1	4.3 (CH_2Te)	
3zj	1672[d]	194.3	−13.6 (CH_3Te)	621.4
3zk	1655	194.7	4.1 (CH_2Te)	801.7
3zl	1670	191.1	−13.1 (CH_3Te)	
3zm	1665	192.0	4.2 (CH_2Te)	831.2
3zn	1670	192.0	4.2 (CH_2Te)	
3zo	1663	198.2	−12.1 (CH_3Te)	
3zp	1658	199.1	5.3 (CH_2Te)	
3zq	1655	198.2	3.9 (CH_2Te)	

[a] Neat. [b] $CDCl_3$. [c] $CDCl_3$. Standard: $(CH_3)_2Te$. [d] Nujol.

Table 8. Spectral Data for *E*-Methyl Benzenecarbochalcogenoates, RCOEMe

		IR (cm−1)[a]	NMR [δ][b]		
R	E	νC═O	^{13}C═O	E	ref
C_6H_5	O	1710	166.8[29]		29
	S	1670[21c]	185.0[29]		21c, 29
	Se	1666[30]	194.7[29]	445.6 (^{77}Se)[c,31]	29, 30, 31
	Te	1655	196.8	613.7 (^{125}Te)[d]	this work

[a] Neat. [b] $CDCl_3$. [c] Standard: $(CH_3)_2Se$. [d] Standard: $(CH_3)_2Te$.

positional parameters are listed in Table 9. Selected bond distances and angles are shown in Table 10. As shown, the ester is monomeric and exists in the *Z*-configuration as do common esters[32] and their sulfur isologues.[33−39] The C(7)−O(1) [1.203(4) Å] and C(7)−Te(1) [2.153(3) Å] and C(8)−Te(1) [2.109(5) Å] bond lengths are in the range typically observed in related molecules, indicating C═O double and C−Te single bonds, respectively. Important structural features are (i) the CH_3Te−CO group and phenyl ring are coplanar, although steric repulsion between Te(1) and H(1) is expected to cause substantial twisting of the functional group toward the plane of the aromatic ring due to the large atomic radii of Te, and (ii) the bond angle 92.3(2)° of C(7)−Te(1)−C(8) is less than those (102−108°) of thioesters.[33] Presumably, the C−Se−C angles of *Se*-alkyl aroylates are 95−98°, which are intermediate between those of thio- and telluroesters. This decrease in the bond angle is consistent with the order of the calculated angles for dimethyl chalcogenides (CH_3ECH_3, E= S, Se, Te).[40]

In conclusion, a series of sodium and potassium tellurocarboxylates were prepared in moderate to good yields. The

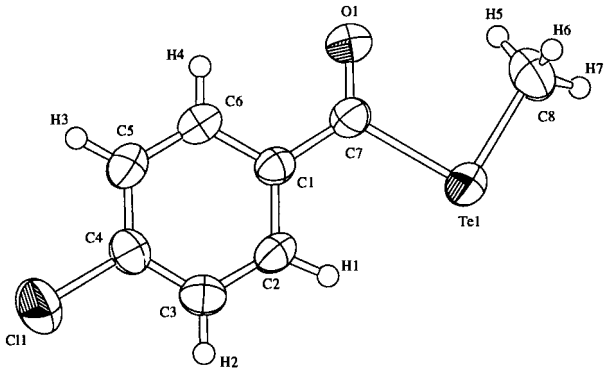

Figure 2. ORTEP drawing of *Te*-methyl 4-chlorobenzenecarbotelluroate (**3zj**). The atoms are drawn with 50% probability thermal ellipsoids.

Table 9. Crystallographic Data for **3zj**

empirical formula	C_8H_7ClOTe
fw	282.20
cryst dimens (mm)	0.57 × 0.40 × 0.40
cryst color	yellow
cryst syst	monoclinic
unit system	
a (Å)	5.975(3)
b (Å)	14.517(2)
c (Å)	10.617(3)
β (deg)	92.74(3)
vol of unit cell (Å^3)	919.8(4)
space group	$P2_1/a$ (No. 14)
Z value	4
D_{calc} (g/cm^3)	2.038
μ(Mo Kα) (cm^{-1})	34.65
temp (°C)	23.0
$\lambda_{MoK\alpha}$ (Å)	0.71069
$2\theta_{max}$ (deg)	55.0
no. of reflns measd	2407
no. of reflns obsd [$I > 3\sigma(I)$]	1797
no. of variables	117
residuals R;[a] R_w[b]	0.033; 0.046
goodness of fit indicator	1.71

[a] $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$. [b] $R_w = [\Sigma(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$, $w = [\sigma^2(F_o) + p^2(F_o)^2/4]^{-1}$.

Table 10. Selected Bond Distances (Å), Angles (deg), and Torsion Angles (deg) of *Te*-Methyl 4-Chlorobenzenecarbotelluroate **3zj**

Bond Distance			
C7−O1	1.203(4)	C1−C7	1.479(5)
C7−Te1	2.153(3)	C4−Cl1	1.734(4)
C8−Te1	2.109(5)	C8···O1	3.02
Angles			
C1−C7−O1	122.5(3)	C7−C1−C2	122.7(3)
C1−C7−Te1	117.8 (2)	C7−C1−C6	118.8(3)
O1−C7−Te1	119.8(3)	Cl1−C4−C3	118.9(3)
C7−Te1−C8	92.3(2)	Cl1−C4−C5	119.3(3)
Torsion Angles			
C8−Te1−C7−O1	0.3(4)	O1−C7−C1−C2	177.1(4)
C8−Te1−C7−C1	179.6(3)	O1−C7−C1−C6	2.9(5)
Te1−C7−C1−C2	2.2(4)	Cl1−C4−C3−C2	179.8(3)
Te1−C7−C1−C6	177.9(2)	Cl1−C4−C5−C6	180.0(3)

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preparation of the sodium salts is more easier than the preparation of the potassium salts. However, purification by recrystallization appeared to be more difficult for the sodium salts than for the potassium salts. A series of *Te*-alkyl tellurocarboxylates are prepared in good yields by reaction of the sodium and potassium salts with alkali metal iodides. The structure of *Te*-methyl 4-chlorobenzenecarbotelluroate showed a plane.

Experimental Section

The IR spectra were measured on a Perkin Elmer FT-IR 1640. The ^{1}H NMR spectra were recorded on a JEOL JNM-GX-270 (270 MHz) and JEOL JNM-α400 (399.7 MHz) with tetramethylsilane as an internal standard, and the following abbreviations were used; s, singlet; d, doublet; t, triplet; q, quartet; quint, quintet; sext, sextet; sept, septet; m, multiplet. ^{13}C NMR spectra were obtained on a JEOL JNM-GX-270 (67.9 MHz) and JEOL JNM-A400 (100.4 MHz) with methanol-d_4 or chloroform-d_1 as an internal standard. The ^{125}Te NMR spectra were obtained on a JEOL JNM-GX-270 (85.9 MHz) and JEOL JNM-α400 (126.0 MHz) with dimethyl telluride as an external standard. Elemental analyses were performed by the Elemental Analysis Center of Kyoto University.

Materials. Tellurium (pieces), which was powdered prior to use, sodium and potassium metals, methyl, ethyl, propyl, isopropyl, butyl, and 1,1-dimethylethyl iodides, and acetyl, isobutyryl, pivaloyl, octadecanoyl, cyclohexanoyl, 1-adamantanecarbonyl, benzoyl, 2-chlorobenzoyl, and 3-chlorobenzoyl chlorides were purchased from Nacalai Tesque Co. (Kyoto, Japan). 4-Chlorobenzoyl, 1-naphthoyl, 2-naphthoyl, 2-methylbenzoyl, 4-methylbenzoyl, 2-methoxybenzoyl, and 4-methoxybenzoyl chlorides were prepared according to the literature.[41] 3-Chloro-2,6-dimethoxybenzoyl chloride (purity: 99.9%) were supplied from Nippon Soda Co., Ltd. Sodium benzoate was commercial grade [IR (KBr) 1556 (C═O) 1418 (C═O) cm^{-1}; ^{1}H NMR (CD_3OD) δ 7.32−7.38 (m, 3H, Ar), 7.94−7.96 (m, 2H, Ar); ^{13}C NMR (CD_3OD) δ 128.6, 130.2, 131.2, 139.0 (Ar), 175.6 (C═O)]. Acetonitrile was distilled under N_2 from phosphorus pentoxide. Ether was distilled under N_2 from sodium benzophenone ketyl. Hexane was distilled under N_2 from sodium metal. All air-sensitive compounds were prepared and handled under argon.

X-ray Measurements. The measurement was carried out on a Rigaku AFC7R four-circle diffractometer with graphite-monochromated Mo Kα radiation (λ = 0.710 69 Å). All of the structures were solved and refined using the teXsan crystallographic software package on an IRIS Indigo computer. X-ray quality crystals of **3zj** were obtained by recrystallization from ether. The crystal was cut from the grown needles. The crystal was mounted on a glass fiber. Because the sample of **3zj** was unstable in air, the crystal was coated with an epoxy resin. The cell dimensions were determined from a least-squares refinement of the setting diffractometer angles for 25 automatically centered reflections. Three standard reflections were measured every 150 reflections, and no decay was detected. Lorentz and polarization corrections were applied to the data, and empirical absorption corrections (ψ-scans[42]) were also applied. The structure was solved by a direct method using SHELXS-86[43] and expanded using DIRDIF92.[44] Scattering factors for neutral atoms were from Cromer and Waber,[45] and anomalous dispersion[46] was used. The function minimized was $\Sigma w(|F_o| - |F_c|)^2$, and the weighting scheme employed was $w = [\sigma^2(F_o) + p^2(F_o)^2/4]^{-1}$. A full-matrix least-squares refinement was executed with non-hydrogen atoms being anisotropic. The final least-squares cycle included fixed hydrogen atoms at calculated positions of which each isotropic thermal parameter was set to 1.2 times that of the connecting atom.

Sodium and Potassium Tellurides. These alkali metal tellurides were prepared according to the method described in the literature.[47] A mixture of tellurium powder (4.20 g, 33.0 mmol) and sodium metal (1.58 g, 69.0 mmol) was stirred in liquid ammonia (ca. 35 mL) in a Schlenk tube (ϕ 4 cm × 16 cm) at −70 °C for 6 h. Most of the ammonia was evaporated by raising gradually (requiring 2 h) from −70 °C to room temperature (23 °C). A trace of ammonia was removed in vacuo under heating (ca. 100 °C, employing a hair dryer) to give sodium telluride (5.40 g, 31.0 mmol) as a white solid involving a trace of black tellurium. The sodium telluride was stored in a Schlenk tube wrapped with aluminum foil at room temperature.

Synthesis of Sodium (1) and Potassium Tellurocarboxylates (2). The preparation of compounds **1k**, **2a**, and **2f** by method A and that of compounds **1a**, **1k**, **1p**, **2a**, and **2j** by method B are described in detail as typical procedures for the preparation of **1** and **2**. Their yields are shown in Tables 1 and 2. The νC═O bands and ^{13}C═O and ^{125}Te chemical shifts are collected in Table 3.

Sodium Telluroacetate (1a). Method B. A solution of acetyl chloride (0.185 g, 2.36 mmol) in acetonitrile solution (3 mL) was added to a suspension of sodium telluride (0.665 g, 3.83 mmol) in the same solvent (9 mL) at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to white green. The mixture was stirred at the same temperature for 2 h. The black precipitates (NaCl, excess Na_2Te, and black tellurium) were filtered off by the use of an Umkehr filter (G4). Removal of the solvent from the filtrate under reduced pressure (23 °C, 3 Torr) gave 0.322 g (70%) of sodium telluroacetate (**1a**) as a yellow green solid. Washing with ether (7 mL) gave **1a** as pale yellow microcrystals (36%). ^{1}H NMR (CD_3OD): δ 2.53 (s, CH_3). ^{13}C NMR (CD_3OD): δ 55.0 (CH_3), 207.8 (C═O). ^{125}Te NMR (CD_3OD): δ 251.9.

Sodium Ethanecarbotelluroate (1b). Method B: pale yellow microcrystals (59%). ^{1}H NMR (CD_3OD): δ 0.96 (t, J = 7.5 Hz, 3H, CH_3), 2.79 (q, J = 7.5 Hz, 2H, CH_2). ^{13}C NMR (CD_3OD): δ 10.9 (CH_3), 59.9 (CH_2), 213.9 (C═O). ^{125}Te NMR (CD_3OD): δ 196.1.

Sodium Propanecarbotelluroate (1c). Method B: pale yellow microcrystals (56%). ^{1}H NMR (CD_3OD): δ 0.99 (t, J = 7.4 Hz, 3H, CH_3), 1.67 (sext, J = 7.4 Hz, 2H, CH_2), 2.83 (t, J = 7.4 Hz, 2H, CH_2-CO). ^{13}C NMR (CD_3OD): δ 13.4 (CH_3), 20.7 (CH_2), 68.7 (CH_2CO), 213.1 (C═O). ^{125}Te NMR (CD_3OD): δ 211.2.

Sodium 1-Methylethanecarbotelluroate (1d). Method B: pale yellow microcrystals (50%). ^{1}H NMR (CD_3OD): δ 0.94 (d, J = 6.8 Hz, 6H, CH_3), 2.95 (sept, J = 6.8 Hz, 1H, CH). ^{13}C NMR (CD_3OD): δ 19.7 (CH_3), 62.0 (CH), 219.7 (C═O). ^{125}Te NMR (CD_3OD): δ 159.9.

Sodium Butanecarbotelluroate (1e). Method B: pale yellow microcrystals (45%). ^{1}H NMR (CD_3OD): δ 0.88 (t, J = 7.5 Hz, 3H, CH_3), 1.31 (sext, J = 7.5 Hz, 2H, CH_2), 1.53 (quint, J = 7.5 Hz, 2H, CH_2), 2.77 (t, J = 7.5 Hz, 2H, CH_2CO). ^{13}C NMR (CD_3OD): δ 14.2 (CH_3), 22.5, 29.5 (CH_2), 66.5 (CH_2CO), 213.1 (C═O). ^{125}Te NMR (CD_3OD): δ 213.4.

Sodium 1,1-Dimethylethanecarbotelluroate (1f). Method A: dark red oil containing small amount of acetonitrile (80%). ^{1}H NMR (CD_3OD): δ 0.98 (s, CH_3): ^{13}C NMR (CD_3OD): δ 28.9 (CH_3), 55.7 (C−CO), 223.6 (C═O). ^{125}Te NMR (CD_3OD): δ 63.0.

Sodium Pentanecarbotelluroate (1g). Method C. Sodium ethoxide (0.170 g, 2.50 mmol) was added to bis(pentanecarbonyl) telluride (0.815 g, 2.50 mmol) in a mixed solvent of ether/pentane (1:1) at −20 °C, and the mixture was stirred for 2 h. Removal of the solvent under reduced pressure gave 0.512 g (82%) of sodium pentanecarbotelluroate **1g** as orange microcrystals: ^{1}H NMR (CD_3OD): δ 0.89 (t, J = 7.7

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Hz, 3H, C*H*$_3$CH$_2$), 1.33 (m, 4H, CH$_2$CH$_2$), 1.65 (m, 2H, CH$_2$), 2.61 (t, J = 7.7 Hz, 2H, CH$_2$CO). ^{13}C NMR (CD$_3$OD): δ 13.5 (CH$_3$), 23.2, 24.4, 30.7 (CH$_2$), 55.5 (CH$_2$CO), 213.2 (C═O). ^{125}Te NMR (CD$_3$OD): δ 224.9.

Sodium Octadecanecarbotelluroate (1h). Method B: white crystals (59%). ^{1}H NMR (CDCl$_3$): δ 0.90 (t, J = 6.8 Hz, 3H, CH$_3$), 1.28 (m, 30H, CH$_2$), 2.75 (t, J = 7.4 Hz, 2H, CH$_2$CO). ^{13}C NMR (CD$_3$OD): δ 17.9 (CH$_3$), 14.1, 22.7, 25.1, 28.7, 29.3, 29.4, 29.6, 29.7 (CH$_2$), 58.0 (CH$_2$CO), 212.6 (C═O).

Sodium Cyclohexanecarbotelluroate (1i). Method A: dark red oil (75%). ^{1}H NMR (CD$_3$OD): δ 1.2−1.9 (m, 10H, CH$_2$), 2.73 (m, 1H, CH). ^{13}C NMR (CD$_3$OD): δ 26.2 (CH$_3$), 26.8, 24.4, 30.6 (CH$_2$), 55.5 (*C*HCO), 220.8 (C═O).

Sodium 1-Adamantanecarbotelluroate (1j). Method B: pale yellow microcrystals (79%). ^{1}H NMR (CD$_3$OD): δ 1.61−1.69 (m, 6H, CH$_2$), 1.80 (d, J = 2.7 Hz, 6H, CH$_2$), 1.94 (s, 3H, CH). ^{13}C NMR (CD$_3$OD): δ 30.0, 38.1, 41.9, 57.7 (C^1), 225.1 (C═O). ^{125}Te NMR (CD$_3$OD): δ 33.9.

Sodium Benzenecarbotelluroate (1k). Method A. A solution of benzoyl chloride (0.441 g, 3.13 mmol) in tetrahydrofuran solution (4 mL) was added to a suspension of sodium telluride (0.545 g, 3.13 mmol) in the same solvent (9 mL) at 0 °C under an argon atmosphere. The mixture was stirred at the same temperature for 1 h. The color of the solution rapidly changed to dark reddish brown. The black precipitates (NaCl and black tellurium) were filtered off. Removal of the solvent from the filtrate under reduced pressure gave 0.640 g (80%) of sodium benzenecarbotelluroate (**1k**) as a dark red oil. ^{1}H NMR (CD$_3$OD): δ 7.20−8.10 (m, Ar). ^{13}C NMR (CD$_3$OD): δ 209.0 (C═O).

Method B. A solution of benzoyl chloride (0.393 g, 2.80 mmol) in acetonitrile solution (3 mL) was added to a suspension of sodium telluride (0.694 g, 4.00 mmol) in the same solvent (18 mL) at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to dark reddish brown. The mixture was stirred at the same temperature for 2 h. The black precipitates (NaCl, excess Na$_2$Te, and black tellurium) were filtered off. The solvent was removed from the filtrate under reduced pressure (23 °C, 3 Torr). Ether (5 mL) was added to the residue (dark red sticky oil), and the mixture was stirred, followed by the addition of hexane (3 mL). Filtration of the resulting precipitates gave a yellow solid. The solid was dissolved in acetonitrile (2 mL) to give a dark red solution. The solution was filtered and concentrated to ca. 1 mL under reduced pressure. To the concentrate, ether (12 mL) and then hexane (11 mL) were added gradually. Filtration of the crystals gave 0.563 g (79%) of sodium benzenecarbotelluroate (**1k**) as yellow plates. ^{1}H NMR (CD$_3$OD): δ 7.28−8.10 (m, Ar). ^{13}C NMR (CD$_3$OD): δ 128.1, 129.5, 132.1, 151.4 (Ar), 208.8 (C═O). ^{125}Te NMR (CD$_3$OD): δ 224.8.

Sodium 2-Methylbenzenecarbotelluroate (1l). Method B: dark red oil containing a small amount of acetonitrile (98%). ^{1}H NMR (CD$_3$OD): δ 2.32 (s, 3H, CH$_3$), 6.98−7.00 (m, 1H, Ar), 7.10−7.14 (m, 2H, Ar), 7.69−7.73 (m, 1H, Ar). ^{13}C NMR (CD$_3$OD): δ 19.9 (CH$_3$), 125.2, 128.8, 129.0, 129.7,131.0, 155.5 (Ar), 213.1 (C═O). ^{125}Te NMR (CD$_3$OD): δ 394.0.

Sodium 3-Methylbenzenecarbotelluroate (1m). Method A: dark red oil containing a small amount of acetonitrile (82%). ^{1}H NMR (CD$_3$OD): δ 2.35 (s, 3H, CH$_3$), 7.23−7.87 (m, 4H, Ar). ^{13}C NMR (CD$_3$OD): δ 21.3 (CH$_3$), 126.4, 127.7, 129.8, 132.5, 137.5, 150.8 (Ar), 209.2 (C═O).

Sodium 4-Methylbenzenecarbotelluroate (1n). Method B: yellow crystals (78%). ^{1}H NMR (CD$_3$OD): δ 2.26 (s, 3H, CH$_3$), 7.09 (d, J = 8.1 Hz, 2H, Ar), 7.97 (d, J = 8.1 Hz, 2H, Ar). ^{13}C NMR (CD$_3$OD): δ 21.5 (CH$_3$), 128.6, 129.7, 142.7, 148.2 (Ar), 208.2 (C═O). ^{125}Te NMR (CD$_3$OD): δ 197.8; Anal. Calcd for C$_8$H$_7$NaOTe: C, 35.62; H, 2.62. Found: C, 35.32; H, 2.70.

Sodium 2-Methoxybenzenecarbotelluroate (1o). Method B: reddish orange microcrystals (39%). ^{1}H NMR (CD$_3$OD): δ 3.75 (s, 3H, CH$_3$O), 6.81−7.90 (m, Ar). ^{13}C NMR (CD$_3$OD): δ 56.8 (CH$_3$O), 113.2, 120.3, 131.0, 131.7, 144.7, 152.1 (Ar), 208.8 (C═O). ^{125}Te NMR (CD$_3$OD): δ 424.4.

Sodium 4-Methoxybenzenecarbotelluroate (1p). Method B. A solution of 4-methoxybenzoyl chloride (0.311 g, 1.82 mmol) in acetonitrile solution (3 mL) was added to a suspension of sodium telluride (0.610 g, 3.51 mmol) in the solvent (12 mL) at 0 °C under an argon atmosphere. The color of the solution rapidly changed from dark gray to reddish brown. The mixture was stirred at the same temperature for 2 h. The dark gray precipitates (NaCl, excess Na$_2$Te, and black tellurium) were filtered off. Removal of the solvent from the filtrate under reduced pressure (3 Torr) gave a crude yellow wet solid. Ether (8 mL) was added to the wet solid, and the mixture was stirred at room temperature for 30 min. Removal of the resulting precipitates by filtration gave yellow microfine solids. The solids were dissolved in acetonitrile (2 mL). The reulting dark red solution was filtered. Ether (10 mL) was gradually added to the filtrate. Removal of the resulting crystals by filtration gave 0.426 g (82%) of sodium 4-methoxybenzenecarbotelluroate (**1p**) as yellow plates. ^{1}H NMR (CD$_3$OD): δ 3.81 (s, 3H, CH$_3$O), 6.79 (d, J = 9.0 Hz, 2H, Ar), 8.04 (d, J = 5.1 Hz, 2H, Ar). ^{13}C NMR (CD$_3$OD): δ 55.8 (CH$_3$O), 112.9, 131.9, 144.1, 163.8 (Ar), 206.1 (C═O). ^{125}Te NMR (CD$_3$OD): δ 174.9; Anal. Calcd for C$_8$H$_7$NaO$_2$Te: C, 33.63; H, 2.47. Found: C, 33.35; H, 2.45.

Sodium 2-Chlorobenzenecarbotelluroate (1q). Method B: reddish orange oil (88%). ^{1}H NMR (CD$_3$OD): δ 7.0−7.48 (m, 4H, Ar). ^{13}C NMR (CD$_3$OD): δ 124.3, 126.4, 128.8, 129.8, 130.2, 154.9, 208.3 (C═O). ^{125}Te NMR (CD$_3$OD): δ 455.3.

Sodium 3-Chlorobenzenecarbotelluroate (1r). Method B: yellow solid (62%): ^{1}H NMR (CD$_3$OD): δ 7.29 (t, J = 7.9 Hz, 1H, Ar), 7.44 (d, J = 7.9 Hz, 1H, Ar), 7.95 (d, J = 7.9 Hz, 1H, Ar), 8.05 (s, 1H, Ar). ^{13}C NMR (CD$_3$OD): δ 127.4, 129.1, 129.4, 131.5, 133.9, 152.5, 207.1 (C═O). ^{125}Te NMR (CD$_3$OD): δ 259.0.

Sodium 4-Chlorobenzenecarbotelluroate (1s). Method B: Recrystallization from acetonitrile (1 mL) and ether (4 mL) gave yellow plates (59%). ^{1}H NMR (CD$_3$OD): δ 7.18 (d, J = 8.3 Hz, 2H, Ar), 7.89 (d, J = 8.3 Hz, 2H, Ar). ^{13}C NMR (CD$_3$OD): δ 128.1, 131.0, 138.4, 150.0 (Ar), 206.8 (C═O). ^{125}Te NMR (CD$_3$OD): δ 240.7.

Sodium 3-Chloro-2,6-dimethoxybenzenecarbotelluroate (1t). Method B: yellow microcrystals (77%). ^{1}H NMR (CDCl$_3$): δ 3.78 (s, 3H, CH$_3$O), 3.97 (s, 3H, CH$_3$O), 6.70 (d, J = 8.9 Hz, 1H, Ar), 7.15 (d, J = 8.9 Hz, 1H, Ar). ^{13}C NMR (CDCl$_3$): δ 56.8 (CH$_3$O), 61.9 (CH$_3$O) 109.8, 119.6, 128.7, 142.5, 148.9, 152.9 (Ar), 203.4 (C═O). ^{125}Te NMR (CD$_3$OD): δ 501.6.

Sodium 1-Naphthalenecarbotelluroate (1u). Method A: reddish orange oil (78%). ^{1}H NMR (CD$_3$OD): δ 7.42−8.44 (m, 10H, Ar). ^{13}C NMR (CD$_3$OD): δ 124.9, 125.7, 126.4, 126.7, 127.0, 127.4, 128.2, 129.4, 134.6 (Ar), 212.0 (C═O).

Sodium 2-Naphthalenecarbotelluroate (1v). Method B: orange microcrystals (76%). ^{1}H NMR (CD$_3$OD): δ 7.44 (t, J = 8.1 Hz, 1H, Ar), 7.50 (t, J = 8.1 Hz, 1H, Ar), 7.75 (d, J = 8.1 Hz, 1H, Ar), 7.78 (d, J = 8.1 Hz, 1H, Ar), 7.97 (t, J = 8.1 Hz, 1H, Ar), 8.22 (t, J = 8.1 Hz, 1H, Ar), 8.80 (s, 1H, Ar). ^{13}C NMR (CD$_3$OD): δ 125.3, 127.0, 127.4, 128.1, 128.2, 130.0, 131.3, 133.2, 135.9, 147.7 (Ar), 208.8 (C═O). ^{125}Te NMR (CD$_3$OD): δ 231.8.

Potassium Methanecarbotelluroate (2a). Method A. A solution of acetyl chloride (0.109 g, 1.39 mmol) in THF (5 mL) was added to potassium telluride (0.287 g, 1.70 mmol) in the same solvent (9 mL) at 20 °C under an argon atmosphere, and the mixture was stirred at the same temperature for 1 h (the color of the solution quickly changed to yellow). Removal of the black precipitates (black Te and KCl) by filtration and removal of the solvent under reduced pressure gave 0.254 g (87%) of potassium benzenecarbotelluroate **2a** as a yellow oil. IR (Nujol): 3007, 2969, 2888, 2842, 1588 (C═O), 1464, 1407, 1378, 1338, 1093, 929, 804, 579 cm^{-1}. ^{1}H NMR (CD$_3$OD): δ 2.60 (s, CH$_3$). ^{13}C NMR (CD$_3$OD): δ 55.1 (CH$_3$), 207.1 (C═O).

Method B. A solution of acetyl chloride (0.145 g, 1.85 mmol) in acetonitrile solution (3 mL) was added to a suspension of potassium telluride (0.623 g, 3.03 mmol) in the same solvent (8 mL) at 0 °C under an argon atmosphere. The color of the solution rapidly changed from dark gray to dark green. The mixture was stirred at the same temperature for 2 h. The black precipitates (KCl, excess K$_2$Te, and black tellurium) were filtered off. Removal of the solvent from the filtrate under reduced pressure (23 °C, 3 Torr) gave 0.210 g (54%) of potassium methanecarbotelluroate (**2a**) as a pale yellow solid, which was sufficiently pure for use in subsequent esterification. Washing with ether (3 mL) (the mixture was stirred at room temperature for 30 min) gave pale yellow microcrystals (0.128 g, 33%) of **2a**. ^{1}H NMR (CD$_3$-

OD): δ 2.61 (s, CH_3). ^{13}C NMR (CD_3OD): δ 55.1 (CH_3), 207.8 (C═O). ^{125}Te NMR (CD_3OD); δ 250.4.

Potassium Propanecarbotelluroate (2b). Method B: pale yellow microcrystals (66%). ^1H NMR (CD_3OD): δ 0.90 (t, J = 7.4 Hz, 3H, CH_3), 1.58 (sext, J = 7.4 Hz, 2H, CH_2), 2.75 (t, J = 7.4 Hz, 2H, CH_2-CO). ^{13}C NMR (CD_3OD): δ 13.4 (CH_3), 20.6 (CH_2), 68.6 (CH_2CO), 213.3 (C═O). ^{125}Te NMR (CD_3OD): δ 213.8.

Potassium 1-Methylethanecarbotelluroate (2c). Method B: pale yellow crystals (45%). ^1H NMR (CD_3OD): δ 0.97 (d, J = 6.8 Hz, 6H, CH_3), 2.97 (sept, J = 6.8 Hz, 1H, CH). ^{13}C NMR (CD_3OD): δ 19.8 (CH_3), 62.1 (CH), 219.6 (C═O). ^{125}Te NMR (CD_3OD); δ 153.5.

Potassium 1,1-Dimethylethanecarbotelluroate (2d). Method B: pale yellow crystals (55%). ^1H NMR (CD_3OD): δ 1.09 (s, CH_3). ^{13}C NMR (CD_3OD): δ 28.9 (CH_3), 55.7 (C−CO), 223.1 (C═O). ^{125}Te NMR (CD_3OD): δ 61.2.

Potassium Pentanecarbotelluroate (2e). Method A: orange oil (82%). ^1H NMR (CD_3OD): δ 0.88 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 1.27 (m, 4H, CH_2CH_2), 1.55 (m, 2H, CH_2), 2.74 (t, J = 7.7 Hz, 2H, CH_2-CO). ^{13}C NMR (CD_3OD): δ 14.3 (CH_3), 23.4, 27.0, 31.8 (CH_2), 66.8 (CH_2CO), 212.9 (C═O). ^{125}Te NMR (CD_3OD): δ 215.1.

Potassium Benzenecarbotelluroate (2f). Method A. A solution of benzoyl chloride (0.239 g, 1.70 mmol) in THF (5 mL) was added to potassium telluride (0.350 g, 1.70 mmol) in the same solvent (9 mL) at 0 °C under an argon atmosphere, and the mixture was stirred at the same temperature for 1 h (the color of the solution quickly changed to dark red). Removal of the black precipitates by filtration and removal of the solvent under reduced pressure gave 95% of potassium benzenecarbotelluroate **2f** as a dark red oil: ^1H NMR (CD_3OD): δ 7.27−8.06 (m, Ar). ^{13}C NMR (CD_3OD): δ 128.1, 129.4, 129.5, 130.3 (Ar), 208.6 (C═O).

Method B. A solution of benzoyl chloride (0.201 g, 1.43 mmol) in acetonitrile solution (3 mL) was added to a suspension of potassium telluride (0.438 g, 2.13 mmol) in the same solvent (8 mL) at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to dark reddish brown. The mixture was stirred at the same temperature for 2 h. The black precipitates (KCl, excess of Na_2Te, and black tellurium) were filtered off. The solvent from the filtrate was removed under reduced pressure (23 °C, 3 Torr). Ether (5 mL) was added to the red residue, and the mixture was stirred for 50 min. Removal of the resulting precipitates by filtration gave a red solid. The solid was dissolved in acetonitrile (1.5 mL) to give a dark red solution, which was then filtered and concentrated to ca. 1 mL under reduced pressure. Ether (7 mL) was slowly added to the concentrate. Removal of the resulting crystals by filtration gave 0.213 g (55%) of potassium benzenecarbotelluroate (**2f**) as red plates. ^1H NMR (CD_3OD): δ 7.25−8.01 (m, Ar). ^{13}C NMR (CD_3OD): δ 128.1, 129.3, 132.2, 150.9 (Ar), 208.9 (C═O). ^{125}Te NMR (CD_3OD): δ 220.0.

Potassium 2-Methylbenzenecarbotelluroate (2g). Method A: dark red oil (containing a small amount of acetonitrile) (90%). ^1H NMR (CD_3OD): δ 2.32 (s, 3H, CH_3), 6.71−7.72 (m, Ar). ^{13}C NMR (CD_3OD): δ 20.0 (CH_3), 125.3, 129.1, 129.9, 131.1, 155.8 (Ar), 213.0 (C═O). ^{125}Te NMR (CD_3OD): δ 390.8.

Potassium 4-Methylbenzenecarbotelluroate (2h). Method B: red crystals (56%). ^1H NMR (CD_3OD): δ 2.27 (s, 3H, CH_3), 7.09 (d, J = 7.7 Hz, 2H, Ar), 7.94 (d, J = 7.7 Hz, 2H, Ar). ^{13}C NMR (CD_3OD): δ 21.5 (CH_3), 128.7, 129.7, 142.8, 148.4 (Ar), 208.0 (C═O). ^{125}Te NMR (CD_3OD): δ 197.3.

Potassium 2-Methoxybenzenecarbotelluroate (2i). Method B: dark red crystals (61%). ^1H NMR (CD_3OD): δ 3.77 (s, 3H, CH_3O), 6.83−7.75 (m, Ar). ^{13}C NMR (CD_3OD): δ 56.3 (CH_3O), 112.8, 120.3, 131.0, 131.1, 145.4, 152.1 (Ar), 208.6 (C═O). ^{125}Te NMR (CD_3OD): δ 413.1.

Potassium 4-Methoxybenzenecarbotelluroate (2j). Method B. A solution of 4-methoxybenzoyl chloride (0.215 g, 1.26 mmol) in acetonitrile solution (4 mL) was added to a suspension of potassium telluride (0.396 g, 1.92 mmol) in the same solvent (7 mL) in a 30 mL round bottom flask at 0 °C under an argon atmosphere. The color of the solution rapidly changed from dark gray to dark reddish brown. The mixture was stirred at the same temperature for 2 h. The black precipitates (KCl, excess K_2Te, and black tellurium) were filtered off by the use of a glass filter (G4). The solvent was removed from the filtrate under reduced pressure (23 °C, 3 Torr). Washing the residue with ether (5 mL) gave an orange solid. The solid was dissolved in acetonitrile (2 mL). The resulting dark red solution was filtered. Ether (8 mL) was slowly added to the filtrate. Removal of the resulting crystals by filtration gave 0.236 g (62%) of potassium 4-methoxybenzenecarbotelluroate (**2j**) as orange plates. ^1H NMR (CD_3OD): δ 3.79 (s, 3H, CH_3O), 6.83 (d, J = 8.9 Hz, 2H, Ar), 8.10 (d, J = 8.9 Hz, 2H, Ar). ^{13}C NMR (CD_3OD): δ 56.1 (CH_3O), 113.0, 131.8, 143.8, 163.8 (Ar), 206.1 (C═O). ^{125}Te NMR (CD_3OD): δ 167.6. Anal. Calcd for C_8H_7KOTe: C, 31.83; H, 2.34. Found: C, 31.78; H, 2.35.

Potassium 4-Chlorobenzenecarbotelluroate (2k). Method B. Recrystallization from acetonitrile (1 mL) and ether (6 mL) gave red crystals (59%). ^1H NMR (CD_3OD): δ 7.30 (d, J = 8.8 Hz, 2H, Ar), 8.06 (d, J = 8.8 Hz, 2H, Ar). ^{13}C NMR (CD_3OD): δ 128.0, 130.8, 138.3, 149.3 (Ar), 207.1 (C═O). ^{125}Te NMR (CD_3OD): δ 237.7. Anal. Calcd for $C_7H_4ClKOTe$: C, 27.45; H, 1.32. Found: C, 27.25; H, 1.39.

Synthesis of *Te*-Alkyl Tellurocarboxylates (3). The preparation of **3f** and **3h** by method A and the of **3a**, **3p**, and **3zj** by method B are described in detail as typical procedures. Their yields are shown in Table 6. The IR and ^1H and ^{13}C NMR spectra of **3** prepared by using the sodium salts **1** were exactly consistent with those of the authentic samples prepared by the reaction of the corresponding potassium salts **2** with alkyl iodides. Their νC═O bands and ^{13}C═O and ^{125}Te chemical shifts are collected in Table 7.

***Te*-Methyl Methanecarbotelluroate (3a). Method B.** A solution of acetyl chloride (0.610 g, 7.77 mmol) in acetonitrile solution (4 mL) was added to a suspension of sodium telluride (1.931 g, 11.12 mmol) in the same solvent (9 mL) in a 50 mL round bottom flask at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to dark green. The mixture was stirred at room temperature for 2 h. The black precipitates (NaCl, excess Na_2Te, and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent from the filtrate under reduced pressure (23 °C, 3 Torr) gave 1.100 g (73%) of sodium methanecarbotelluroate (**1a**) as a dark green solid. Methyl iodide (15 mL, 240 mmol) was added to the solid at 0 °C. The mixture was stirred at the same temperature in the dark (the flask was wrapped with aluminum foil) for 1 h. Ether (15 mL) was added, and mixture was stirred at room temperature for 20 min. The gray precipitates (NaI and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent under reduced pressure (23 °C, 40 Torr) gave 0.907 g (63%) of *Te*-methyl methanecarbotelluroate (**3a**) as an orange liquid, which was sufficiently pure for use in subsequent experiments. An analytical sample was obtained by distillation under reduced pressure (0.410 g, 28%) as an orange yellow liquid. Bp: 42−48 °C/30 Torr. ^1H NMR ($CDCl_3$): δ 2.05 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 20.7 Hz), 2.40 (s, CH_3CO). ^{13}C NMR ($CDCl_3$): δ −14.1 (CH_3Te, $^1J_{C-Te}$ = 159 Hz), 42.7 (CH_3C), 196.8 (C═O). ^{125}Te NMR ($CDCl_3$): δ 663.5. UV/vis (Et_2O): 286, 238 nm. Anal. Calcd for C_3H_6OTe: C, 19.41; H, 3.26. Found: C, 19.42; H, 3.10.

***Te*-Ethyl Methanecarbotelluroate (3b). Method B:** orange liquid (39%). Bp: 58−60 °C/30 Torr. ^1H NMR ($CDCl_3$): δ 1.67 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 2.46 (s, 3H, CH_3CO), 2.85 (q, J = 7.7 Hz, 2H, CH_2). ^{13}C NMR ($CDCl_3$): δ 3.6 (CH_2Te), 17.7 (CH_3), 43.2 (CH_3CO), 197.0 (C═O). Anal. Calcd for C_4H_8OTe: C, 24.06; H, 4.04. Found: C, 23.78; H, 3.85.

***Te*-Methyl Ethanecarbotelluroate (3c). Method B:** orange liquid (56%). ^1H NMR ($CDCl_3$): δ 1.15 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 2.11 (s, 3H, CH_3Te), 2.63 (q, J = 7.7 Hz, 2H, CH_2). ^{13}C NMR ($CDCl_3$): δ −15.3 (CH_3Te), 9.0 (CH_3), 42.7 (CH_2), 202.3 (C═O). MS (CI) *m*/*z*: 203, 201, 199 (M + 1).

***Te*-Ethyl Ethanecarbotelluroate (3d). Method B:** orange liquid (73%). ^1H NMR ($CDCl_3$): δ 1.06 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 1.61 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 2.56 (q, J = 7.7 Hz, 2H, CH_2CO), 2.78 (q, J = 7.7 Hz, 2H, CH_2Te). ^{13}C NMR ($CDCl_3$): δ 2.7 (CH_2Te), 9.0 (CH_3), 17.7 (CH_3), 49.2 (CH_2CO), 203.0 (C═O). MS (CI) *m*/*z*: 217, 215, 213 (M + 1), 57 (C_2H_5CO).

***Te*-Methyl Propanecarbotelluroate (3e). Method A using salt 1c:** orange yellow liquid (85%). Bp: 69−72 °C/15 Torr. ^1H NMR ($CDCl_3$): δ 0.98 (t, J = 7.4 Hz, 3H, CH_3CH$_2$), 1.68 (sext, J = 7.4 Hz, 2H, CH_2), 2.10 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 21.2 Hz), 2.60 (t, J = 7.4 Hz, 2H, CH_2CO). ^{13}C NMR ($CDCl_3$): δ −14.8 (CH_3Te, $^1J_{C-Te}$ = 160 Hz),

13.1 (CH_3CH_2), 18.5 (CH_3CH_2), 57.2 (CH_2CO), 202.0 (C═O). ^{125}Te NMR ($CDCl_3$): δ 633.0.

***Te*-Methyl 1-Methylethanecarbotelluroate (3f). Method A using salt 1d.** Methyl iodide (10 mL, 160 mmol) was added to sodium 1-methylethanecarbotelluroate **1d** (0.978 g, 4.41 mmol) at 0 °C. The mixture was stirred at the same temperature in the dark (the flask was wrapped with aluminum foil) for 1 h. Ether (15 mL) was added, and the mixture was stirred at room temperature for 15 min. The white gray precipitates (NaI and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent under reduced pressure (23 °C, 40 Torr) gave 0.722 g (77%) of *Te*-methyl 1-methylethanecarbotelluroate (**3f**) as an orange liquid, which was sufficiently pure for use in subsequent experiments. Distillation of the liquid under reduced pressure gave 0.407 g (43%) of chemically pure **3f** as orange yellow liquid. Bp: 57−59 °C/16 Torr. ^{1}H NMR ($CDCl_3$): δ 1.07 (d, J = 6.9 Hz, 6H, CH_3), 1.99 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.2 Hz), 2.56 (sept, J = 6.9 Hz, 1H, CH). ^{13}C NMR ($CDCl_3$): δ −15.3 (CH_3Te, $^1J_{C-Te}$ = 160 Hz), 18.2 (CH_3), 52.2 (CH), 208.5 (C═O). ^{125}Te NMR ($CDCl_3$): δ 579.9. UV/vis (Et_2O): 288, 241 nm. Anal. Calcd for $C_5H_{10}OTe$: C, 28.10; H, 4.72. Found: C, 28.40; H, 4.85.

***Te*-Methyl Butanecarbotelluroate (3g). Method A using salt 1e:** Orange yellow liquid (76%). Bp: 78−81 °C/12 Torr. ^{1}H NMR ($CDCl_3$): δ 0.91 (t, J = 7.4 Hz, 3H, CH_3CH$_2$), 1.38 (sext, J = 7.4 Hz, 2H, CH_2), 1.63 (quint, J = 7.4 Hz, 2H, CH_2), 2.10 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 19.5 Hz), 2.62 (t, J = 7.4 Hz, 2H, CH_2CO). ^{13}C NMR ($CDCl_3$): δ −14.9 (CH_3Te, $^1J_{C-Te}$ = 160 Hz), 13.6 (CH_3CH_2), 21.7, 27.0 (CH_2), 55.1 (CH_2CO), 201.9 (C═O). ^{125}Te NMR ($CDCl_3$): δ 631.0. Anal. Calcd for $C_6H_{12}OTe$: C, 31.64; H, 5.31. Found: C, 31.78; H, 5.44.

***Te*-Methyl 1,1-Dimethylethanecarbotelluroate (3h). Method A using salt 1f.** Methyl iodide (5 mL, 80 mmol) was added to 0.455 g (1.93 mmol) of sodium 1,1-dimethylethanecarbotelluroate (**1f**) at 0 °C. The mixture was stirred at the same temperature in the dark (the flask was wrapped with aluminum foil) for 1 h. Ether (10 mL) was added, and the mixture was stirred at room temperature for 10 min. The white gray precipitates (NaI and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent under reduced pressure (23 °C, 3 Torr) gave 0.373 g (85%) of *Te*-methyl 1,1-dimethylethanecarbotelluroate (**3h**) as an orange liquid, which was sufficiently pure for use in subsequent experiments. Distillation of the liquid under reduced pressure gave 0.150 g (34%). bp: 80−82 °C/70 Torr. ^{1}H NMR ($CDCl_3$): δ 1.06 (s, 9H, CH_3C) 1.95 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.9 Hz). ^{13}C NMR ($CDCl_3$): δ −15.5 (CH_3Te, $^1J_{C-Te}$ = 162 Hz), 26.1 (CH_3C), 52.3 (C−CO), 211.6 (C═O). ^{125}Te NMR ($CDCl_3$): δ 547.9. UV/vis (Et_2O): 290, 240 nm. Anal. Calcd for $C_6H_{12}OTe$: C, 31.64; H, 5.31. Found: C, 31.84; H, 5.37.

***Te*-Ethyl 1,1-Dimethylethanecarbotelluroate (3i). Method B:** orange liquid (70%). ^{1}H NMR ($CDCl_3$): δ 1.13 (s, 9H, CH_3C), 1.65 (t, J = 7.7 Hz, 3H, CH_3CH$_2$Te), 2.88 (q, J = 7.7 Hz, 2H, CH_2Te). ^{13}C NMR ($CDCl_3$): δ 2.0 (CH_2Te), 17.6 (CH_3), 26.2 (CH_3C), 52.6 (C−CO), 212.2(C═O).

***Te*-Methyl Pentanecarbotelluroate (3j). Method B:** orange liquid. Yield: 99%. Bp: 78−80 °C/8 Torr. ^{1}H NMR ($CDCl_3$): δ 0.89 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 1.33 (m, 4H, CH_2CH_2), 1.65 (m, 2H, CH_2), 2.10 (s, 3H, CH_3Te), 2.61 (t, J = 7.7 Hz, 2H, CH_2CO). ^{13}C NMR ($CDCl_3$): δ −15.0 (CH_3Te), 13.7 (CH_3), 22.2, 24.6, 30.7 (CH_2), 55.3 (CH_2CO), 202.7 (C═O).

***Te*-Ethyl Pentanecarbotelluroate (3k). Method B:** orange liquid (83%). Bp: 92−94 °C/8 Torr. ^{1}H NMR ($CDCl_3$): δ 0.89 (t, J = 7.7 Hz, 3H, CH_3CH$_2$CH$_2$), 1.32 (m, 4H, CH_2CH_2), 1.64 (m, 2H, CH_2), 1.68 (t, J = 7.7 Hz, 3H, CH_3CH$_2$Te), 2.62 (t, J = 7.7 Hz, 2H, CH_2CO), 2.84 (q, J = 7.7 Hz, 2H, CH_2Te). ^{13}C NMR ($CDCl_3$): δ 2.7 (CH_2Te), 17.7 (CH_3), 13.6, 22.1, 24.5, 30.6 (CH_2, CH_3), 55.8 (CH_2CO), 202.1 (C═O). ^{125}Te NMR (CD_3OD): δ 814.1; MS (CI): *m*/*z* 260, 258, 256 (M + 1), 99 ($C_5H_{11}CO$). Anal. Calcd for $C_8H_{18}OTe$: C, 37.56; H, 6.31. Found: C, 37.29; H, 6.45.

***Te*-Ethyl Octadecanecarbotelluroate (3l). Method A using sodium salt 1h:** yellow crystals (59%). Mp: 30−31 °C. ^{1}H NMR ($CDCl_3$): δ 0.88 (t, J = 7.7 Hz, 3H, CH_3), 1.26 (m, 30H, CH_2), 1.68 (t, J = 7.7 Hz, 3H, CH_3CH$_2$Te), 2.61 (t, J = 7.7 Hz, 2H, CH_2CO), 2.85 (q, J = 7.7 Hz, 2H, CH_2Te). ^{13}C NMR ($CDCl_3$): δ 2.9 (CH_2Te), 17.9 (CH_3), 14.1, 22.7, 25.1, 28.7, 29.3, 29.4, 29.6, 29.7, 31.9 (CH_2, CH_3), 56.0 (CH_2CO), 202.6 (C═O). MS (CI) *m*/*z* 425, 423, 421, 420 (M + 1), 407, 405, 403 ($C_{17}H_{35}TeC_2H_5$), 268 ($C_{17}H_{35}CO^+$).

***Te*-Methyl Cyclohexanecarbotelluroate (3m). Method A using sodium salt 1i:** orange yellow liquid (80%). Bp: 91−92/5 Torr °C. ^{1}H NMR ($CDCl_3$): δ 1.16−1.46 (m, 5H, cyclohexane), 1.63−1.67 (m, 1H, cyclohexane), 1.77−1.81 (m, 2H, cyclohexane), 1.93−1.97 (m, 2H, cyclohexane), 2.06 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.0 Hz), 2.35−2.43 (m, 1H, CH). ^{13}C NMR ($CDCl_3$): δ −15.4 (CH_3Te, J_{C-Te} = 160.4 Hz), 25.1, 25.7, 28.8 (CH_2), 61.5 (CH), 207.6 (C═O). ^{125}Te NMR ($CDCl_3$): δ 588.3; Anal. Calcd for $C_8H_{14}OTe$: C, 37.86; H, 5.56. Found: C, 38.04; H, 5.49.

***Te*-Methyl 1-Adamantanecarbotelluroate (3n). Method A using sodium salt 1j:** orange yellow liquid (79%). ^{1}H NMR ($CDCl_3$): δ 1.61−1.69 (q, J = 11.1 Hz, 6H, Ad), 1.75 (d, J = 2.4 Hz, 6H, Ad), 1.93 (s, $^2J_{H-Te}$ = 22.9 Hz, 3H, CH_3Te). ^{13}C NMR ($CDCl_3$): δ −16.2 (CH_3Te, J_{C-Te} = 161.9 Hz), 28.0, 36.3, 38.7, 54.3 (Ad), 212.5 (C═O). ^{125}Te NMR ($CDCl_3$): δ 540.4.

***Te*-2-methylpropyl 1-Adamantanecarbotelluroate (3o). Method A using sodium salt 1j:** pale yellow oil (90%). ^{1}H NMR ($CDCl_3$): δ 0.97 (d, J = 6.6 Hz, 6H, CH_3), 1.71−2.07 (m, 14H, adamantyl), 1.83 (m, J = 6.6 Hz, 1H, CH), 2.81 (d, J = 6.6 Hz, 2H, CH_2), 3.14 (d, J = 5.9 Hz, 1H, CH). ^{13}C NMR ($CDCl_3$): δ 20.7 (CH_2), 22.5 (bridgehead), 24.1 (CH_3), 28.1 (adamantyl methylene), 29.9 (CH), 36.5 (adamantyl), 38.9 (adamantyl), 212.7 (C═O). MS (CI): *m*/*z* 351, 349, 347, 346, 345 (M + 1), 163 ($C_{10}H_{15}CO^+$), 135 ($C_{10}H_{15}^+$).

***Te*-Methyl Benzenecarbotelluroate (3p). Method B.** A solution of benzoyl chloride (0.647 g, 4.60 mmol) in acetonitrile solution (3 mL) was added to a suspension of sodium telluride (1.138 g, 6.56 mmol) in the same solvent (22 mL) in a 50 mL round bottom flask at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to dark reddish brown. The mixture was stirred at room temperature for 2 h. The black precipitates (NaCl, excess Na_2Te, and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent from the filtrate under reduced pressure (23 °C, 3 Torr) gave 0.964 g (3.77 mmol) of sodium benzenecarbotelluroate (**1k**) as a deep yellow solid. Methyl iodide (10 mL, 160 mmol) was added to the solid at 0 °C. The mixture was stirred at the same temperature in the dark (the flask was wrapped with aluminum foil) for 1 h. Ether (15 mL) was added, and mixture was stirred at room temperature for 15 min. The white gray precipitates (NaI and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent under reduced pressure (23 °C, 3 Torr) gave 0.819 g (72%) of *Te*-methyl benzenecarbotelluroate (**3p**) as an orange yellow liquid. Distillation of the liquid under reduced pressure gave 0.470 g (41%) of chemically pure **3p** as an orange yellow liquid. Bp: 64−67 °C/0.1 Torr. ^{1}H NMR ($CDCl_3$): δ 2.24 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.7 Hz), 7.38−7.74 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ −14.0 (CH_3Te, $^1J_{C-Te}$ = 160 Hz), 126.7, 128.8, 133.6, 142.7 (Ar), 195.7 (C═O). ^{125}Te NMR ($CDCl_3$): δ 613.7. UV/vis (Et_2O): 257, 340 nm. Anal. Calcd for C_8H_8OTe: C, 38.78; H, 3.25. Found: C, 38.91; H, 3.28.

***Te*-Ethyl Benzenecarbotelluroate (3q). Method B:** orange liquid (67%). Bp: 76−78 °C/0.2 Torr (lit.[21] bp 80.3 °C/0.3 Torr). ^{1}H NMR ($CDCl_3$): δ 1.65 (t, J = 7.7 Hz, 3H, CH_3), 2.93 (q, J = 7.7 Hz, 2H, CH_2Te), 7.16−7.30 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.6 (CH_2Te), 17.7 (CH_3), 126.9, 128.8, 133.6, 143.1 (Ar), 196.4 (C═O). ^{125}Te NMR (CD_3OD): δ 793.6. MS (CI): *m*/*z* 265, 263, 261 (M + 1), 105 (C_6H_5-CO). Anal. Calcd for $C_9H_{10}OTe$: C, 41.30; H, 3.85. Found: C, 41.17; H, 3.81.

***Te*-Propyl Benzenecarbotelluroate (3r). Method B using sodium salt 1k:** orange liquid (80%). Bp: 82−84 °C/0.1 Torr (lit.[21] 105−106 °C/0.2 Torr). ^{1}H NMR ($CDCl_3$): δ 1.02 (t, J = 7.7 Hz, 3H, CH_3), 1.89 (m, 2H, CH_2), 3.06 (t, J = 7.7 Hz, 2H, CH_2Te), 7.39−7.76 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ 14.0 (CH_2Te), 16.8 (CH_2), 25.2 (CH_3), 126.9, 128.8, 133.6, 143.2 (Ar), 196.1 (C═O). Anal. Calcd for $C_{10}H_{12}OTe$: C, 43.55; H, 4.39. Found: C, 43.35; H, 4.35.

***Te*-Allyl Benzenecarbotelluroate (3s). Method B:** orange liquid (71%). Bp: 78−80 °C/0.1 Torr. ^{1}H NMR ($CDCl_3$): δ 3.77 (d J = 7.7 Hz, 2H, CH_2Te), 4.86 (dd, J = 11.3, 1.1 Hz, 1H, H_2C═C), 5.16 (dd, J = 17.9, 1.1 Hz, 1H, H_2C═C), 6.07 (m, 1H, CH═CH_2), 7.37−7.72 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ 13.5 (CH_2Te), 115.8, 126.9, 128.8,

133.7, 136.5, 142.7 (Ar, CH_2═CH), 195.9 (C═O). MS (CI): *m*/*z* 276, 274, 272 (M + 1), 105 (C_6H_5CO).

***Te*-2-Propargyl Benzenecarbotelluroate (3t). Method B:** orange liquid (68%). Bp: 76−80 °C/0.1 Torr. ^{1}H NMR ($CDCl_3$): δ 2.05 (s, 1H, HC≡C), 4.60 (s, 2H, CH_2Te), 7.11−7.54 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ −8.1 (CH_2Te), 70.3 (*C*≡CH), 83.1 (H*C*≡C), 126.7, 126.9, 129.0, 134.1 (Ar), 209.0 (C═O). MS (CI): *m*/*z* 275, 273, 271 (M + 1), 105 (C_6H_5CO).

***Te*-2-Hydroxyethyl Benzenecarbotelluroate (3u). Method B:** orange oil (87%). ^{1}H NMR ($CDCl_3$): δ 2.67 (s, 1H, OH), 3.23 (t, J = 7.7 Hz, 2H, CH_2Te), 3.99 (t, J = 7.7 Hz, 2H, CH_2O), 7.43−7.75 (m, 5H, Ar). ^{13}C NMR ($CDCl_3$): δ 14.9 (CH_2Te), 63.6 (CH_2O), 127.0, 128.0, 133.9, 1423.7 (Ar), 196.2 (C═O). MS (CI): *m*/*z* 281, 279, 277 (M + 1), 263 (M − OH), 105 (C_6H_5CO).

***Te*-Methyl 2-Methylbenzenecarbotelluroate (3v). Method A using sodium salt 1l:** orange liquid (86%). Bp: 77−78 °C/4 Torr. Yellow crystals: mp 15−20 °C. ^{1}H NMR ($CDCl_3$): δ 2.11 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.4 Hz), 2.34 (s, 3H, CH_3Ar), 7.04−7.56 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ −12.8 (CH_3Te, $^1J_{C-Te}$ = 162 Hz), 20.7 (CH_3Ar), 126.1, 129.3, 131.6, 131.8, 134.0, 142.6 (Ar), 198.0 (C═O). ^{125}Te NMR ($CDCl_3$): δ 657.9. UV/vis (Et_2O): 332, 288, 253 nm. Anal. Calcd for $C_9H_{10}OTe$: C, 41.29; H, 3.85. Found: C, 41.34; H, 3.67.

***Te*-Ethyl 2-Methylbenzenecarbotelluroate (3w). Method A using sodium salt 1l:** yellow liquid (56%). ^{1}H NMR ($CDCl_3$): δ 1.66 (t, J = 7.7 Hz, 3H, CH_3), 2.35 (s, 3H, CH_3Ar), 2.89 (q, J = 7.7 Hz, 2H, CH_2), 7.1−7.5 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 4.7 (CH_2Te), 17.7 (CH_3), 20.7 (CH_3Ar), 126.1, 129.5, 131.6, 131.7, 134.0, 143.1 (Ar), 198.8 (C═O). MS (CI): *m*/*z* 279, 278, 277, 275, 274, 273 (M + 1), 119 ($CH_3C_6H_4CO^+$).

***Te*-Ethyl 3-Methylbenzenecarbotelluroate (3x). Method A using sodium salt 1m:** orange liquid (53%). ^{1}H NMR ($CDCl_3$): δ 1.74 (t, J = 7.7 Hz, 3H, CH_3), 2.40 (s, 3H, CH_3Ar), 3.01 (q, J = 7.7 Hz, 2H, CH_2Te), 7.27−7.52 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.5 (CH_2Te), 17.7 (CH_3), 21.2 (CH_3Ar), 124.3, 127.2, 128.7, 134.4, 138.8, 143.2 (Ar), 196.5 (C═O). MS (CI): *m*/*z* 279, 278, 277, 275, 274, 273 (M + 1), 119 ($CH_3C_6H_4CO^+$).

***Te*-Methyl 4-Methylbenzenecarbotelluroate (3y). Method A using sodium salt 1n:** Orange liquid (95%). Bp: 76−78 °C/0.1 Torr. ^{1}H NMR ($CDCl_3$): δ 2.14 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.5 Hz), 2.25 (s, 3H, CH_3Ar), 7.12 (d, J = 8.3 Hz, 2H, Ar), 7.53 (d, J = 8.5 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ −14.2 (CH_3Te, $^1J_{C-Te}$ = 160 Hz), 21.7 (CH_3-Ar), 126.9, 129.5, 140.4, 144.7 (Ar), 194.8 (C═O). ^{125}Te NMR ($CDCl_3$): δ 601.6. UV/vis (Et_2O): 340, 260 nm. MS (CI): *m*/*z* 265, 263, 261 (M + 1), 119 ($CH_3C_6H_4CO$). Anal. Calcd for $C_9H_{10}OTe$: C, 41.29; H, 3.85. Found: C, 42.28; H, 3.86.

***Te*-Ethyl 4-Methylbenzenecarbotelluroate (3z). Method B:** orange liquid (73%). Bp: 86−90 °C/0.25 Torr. ^{1}H NMR ($CDCl_3$): δ 1.61 (t, J = 7.7 Hz, 3H, CH_3), 2.20 (s, 3H, CH_3Ar), 2.88 (q, J = 7.7 Hz, 2H, CH_2Te), 7.04−7.51 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.3 (CH_2Te), 17.7 (CH_3), 21.6 (CH_3Ar), 126.9, 129.3, 140.5, 144.5 (Ar), 195.3 (C═O). ^{125}Te NMR (CD_3OD): δ 781.8. MS (CI): *m*/*z* 279, 277, 275 (M + 1), 119 ($CH_3C_6H_4CO$). Anal. Calcd for $C_{10}H_{12}OTe$: C, 43.55; H, 4.39. Found: C, 43.34; H, 4.48.

***Te*-Methyl 2-Methoxybenzenecarbotelluroate (3za). Method A using sodium salt 1o:** orange liquid (85%). Bp: 101−103 °C/0.1 Torr. Yellow crystals: mp 20−25 °C. ^{1}H NMR ($CDCl_3$): δ 2.05 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 26.6 Hz), 3.95 (s, 3H, CH_3O), 6.97−7.73 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ −13.4 (CH_3Te, $^1J_{C-Te}$ = 165 Hz), 54.9 (CH_3O), 111.9, 120.5, 126.6, 129.5, 134.0, 159.1 (Ar), 189.3 (C═O). ^{125}Te NMR ($CDCl_3$): δ 761.2. UV/vis (Et_2O): 304, 254 nm. MS (CI): *m*/*z* 281, 279, 277 (M + 1), 135 ($CH_3OC_6H_4CO$). Anal. Calcd for $C_9H_{10}O_2Te$: C, 38.92; H, 3.63. Found: C, 38.98; H, 3.39.

***Te*-Ethyl 2-Methoxybenzenecarbotelluroate (3zb). Method B:** orange liquid (85%). Bp: 112−116 °C/0.3 Torr. ^{1}H NMR ($CDCl_3$): δ 1.72 (t, J = 7.7 Hz, 3H, CH_3CH$_2$), 2.84 (q, J = 7.7 Hz, 2H, CH_2Te), 3.94 (s, 3H, CH_3O), 6.95−7.72 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.2 (CH_2Te), 17.0 (CH_3), 55.0 (CH_3O), 112.0, 120.6, 127.0, 130.2, 134.0, 159.0 (Ar), 190.0 (C═O). MS (CI): *m*/*z* 295, 293, 291 (M + 1), 135 ($CH_3OC_6H_4CO$); Anal. Calcd for $C_{10}H_{12}O_2Te$: C, 41.16; H, 4.15. Found: C, 40.75; H, 4.13.

***Te*-Propyl 2-Methoxybenzenecarbotelluroate (3zc). Method B:** Orange liquid (78%). Bp: 112−115 °C/0.2 Torr. ^{1}H NMR ($CDCl_3$): δ 1.01 (t, J = 7.7 Hz, 3H, CH_3), 1.88 (m, 2H, CH_2), 2.87 (t, J = 7.7 Hz, 2H, CH_2Te), 3.94 (s, 3H, CH_3O), 6.95−7.72 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 13.3 (CH_2Te), 16.9 (CH_2), 24.8 (CH_3), 55.0 (CH_3O), 112.0, 120.6, 127.0, 130.1, 133.9, 158.9 (Ar), 189.7 (C═O). MS (CI): *m*/*z* 309, 307, 305 (M + 1), 135 ($CH_3OC_6H_4CO$). Anal. Calcd for $C_{11}H_{14}O_2$-Te: C, 43.20 H, 4.62. Found: C, 43.15; H, 4.68.

***Te*-1-Methylethyl 2-Methoxybenzenecarbotelluroate (3zd). Method A using sodium salt 1o:** yellow liquid (74%). ^{1}H NMR ($CDCl_3$): δ 1.66 (d, J = 7.0 Hz, 6H, CH_3), 3.56 (m, J = 7.0 Hz, 1H, CH), 3.87 (s, 3H, CH_3O), 6.89−7.63 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 19.2 (CH_3), 26.1 (TeCH), 55.1 (CH_3O), 112.1, 120.1, 127.2, 131.0, 134.0, 158.9 (Ar), 191.9 (C═O). MS (CI): *m*/*z* 309, 307, 305 304, 303 (M + 1), 135 ($CH_3OC_6H_4CO^+$).

***Te*-Butyl 2-Methoxybenzenecarbotelluroate (3ze). Method A using sodium salt 1o:** yellow liquid (70%). ^{1}H NMR ($CDCl_3$): δ 0.84 (t, J = 7.3 Hz, 3H, CH_3), 1.33 (m, 2H, CH_2), 1.74 (m, 2H, CH_2), 2.78 (t, J = 7.3 Hz, 2H, CH_2), 3.85 (s, 3H, CH_3O), 6.86−7.63 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 10.6 (CH_2Te), 13.4 (CH_2), 25.5 (CH_3), 33.6 (CH_2), 55.0 (CH_3O), 112.0, 121.6, 127.0, 130.3, 133.9, 159.0 (Ar), 189.8 (C═O). MS (CI): *m*/*z* 323, 318, 317 (M + 1), 135 ($CH_3OC_6H_4CO^+$).

***Te*-Methyl 4-Methoxybenzenecarbotelluroate (3zf). Method A using sodium salt 1p:** orange liquid (85%). Bp: 117−119 °C/2.5 Torr. ^{1}H NMR ($CDCl_3$): δ 2.11 (s, 3H, CH_3Te, $^2J_{H-Te}$ = 22.4 Hz), 3.70 (s, 3H, CH_3O), 6.77 (d, J = 9.0 Hz, 2H, Ar), 7.59 (d, J = 8.8 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ −14.4 (CH_3Te, $^1J_{C-Te}$ = 160 Hz), 55.4 (CH_3O), 113.8, 129.0, 135.6, 163.9 (Ar), 192.7 (C═O). ^{125}Te NMR ($CDCl_3$): δ 584.0. UV/vis (Et_2O): 332, 274, 223 nm. Anal. Calcd for $C_9H_{10}O_2Te$: C, 38.92; H, 3.63. Found: C, 38.77; H, 3.65.

***Te*-Ethyl 4-Methoxybenzenecarbotelluroate (3zg). Method A using sodium salt 1p:** yellow liquid (59%). Bp: 117−119 °C/2.5 Torr. ^{1}H NMR ($CDCl_3$): δ 1.64 (t, J = 7.7 Hz, 3H, CH_3), 2.90 (q, J = 7.7 Hz, 2H, CH_2Te), 3.73 (s, 3H, CH_3O), 6.79 (d, J = 8.8 Hz, 2H, Ar), 7.60 (d, J = 8.8 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.3 (CH_2Te), 17.7 (CH_3), 55.5 (CH_3O), 113.9, 129.2, 136.1, 164.0 (Ar), 193.5 (C═O). MS (CI): *m*/*z* 295, 293, 291, 290, 289 (M + 1), 135 ($CH_3OC_6H_4$-CO^+).

***Te*-Ethyl 2-Chlorobenzenecarbotelluroate (3zh). Method A using sodium salt 1q:** yellow liquid (70%). ^{1}H NMR ($CDCl_3$): δ 1.67 (t, J = 7.7 Hz, 3H, CH_3), 2.91 (q, J = 7.7 Hz, 2H, CH_2Te), 7.21−7.49 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 5.7 (CH_2Te), 17.4 (CH_3), 126.8, 128.2, 128.7, 131.0, 132.3, 142.6 (Ar), 195.2(C═O). MS (CI): *m*/*z* 301, 299, 297, 295 (M + 1), 139 (2-$ClC_6H_4CO^+$).

***Te*-Ethyl 3-Chlorobenzenecarbotelluroate (3zi). Method A using sodium salt 1r:** yellow liquid (63%). ^{1}H NMR ($CDCl_3$): δ 1.74 (t, J = 7.7 Hz, 3H, CH_3), 3.04 (q, J = 7.7 Hz, 2H, CH_2Te), 7.36−7.69 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 4.3 (CH_2Te), 17.6 (CH_3), 125.2, 126.4, 130.1, 133.4, 135.2, 144.7 (Ar), 195.1 (C═O). MS (CI): *m*/*z* 301, 299, 297, 295 (M + 1), 139 (3-$ClC_6H_4CO^+$).

***Te*-Methyl 4-Chlorobenzenecarbotelluroate (3zj). Method B using sodium salt 1s:** A solution of 4-chlorobenzoyl chloride (0.845 g, 4.83 mmol) in acetonitrile solution (3 mL) was added to a suspension of sodium telluride (1.221 g, 7.03 mmol) in the same solvent (21 mL) in a 50 mL round bottom flask at 0 °C under an argon atmosphere. The color of the solution rapidly changed from gray to dark reddish brown. The mixture was stirred at room temperature for 2 h. The black precipitates (NaCl, excess Na_2Te, and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent from the filtrate under reduced pressure (23 °C, 3 Torr) gave 1.221 g (87%) of sodium 4-chlorobenzenecarbotelluroate (**1s**) as an orange solid. Methyl iodide (20 mL, 320 mmol) was added to the solid at 0 °C. The mixture was stirred at the same temperature in the dark (the flask was wrapped with aluminum foil) for 1 h. Ether (10 mL) was added, and mixture was stirred at room temperature for 15 min. The white gray precipitates (NaI and black tellurium) were filtered off with a glass filter (G4). Removal of the solvent under reduced pressure (23 °C, 3 Torr) gave 1.151 g (84%) of crude *Te*-methyl 4-chlorobenzenecarbotelluroate (**3zj**) as a yellow green solid, which was spectroscopically pure. Recrystallization from ether (8 mL) gave yellow needles on standing in a freezer at ca. −20 °C (on standing at room temperature, recrystallization gave

yellow plates). Mp: 81−83 °C. ^{1}H NMR ($CDCl_3$): δ 2.27 (s, 3H, CH_3-Te, $^2J_{H-Te}$ = 22.7 Hz), 7.40 (d, J = 8.4 Hz, 2H, Ar), 7.68 (d, J = 8.4 Hz, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ −13.6 (CH_3Te, $^1J_{C-Te}$ = 159 Hz), 128.0, 129.2, 140.1, 141.3 (Ar), 194.3 (C═O). ^{125}Te NMR ($CDCl_3$): δ 621.4. UV/vis (Et_2O): 342, 263 nm. Anal. Calcd for C_8H_7ClOTe: C, 34.05; H, 2.50. Found: C, 34.22; H, 2.52.

***Te*-Ethyl 4-Chlorobenzenecarbotelluroate (3zk). Method A using sodium salt 1s:** orange liquid (68%). Bp: 90−93 °C/0.1 Torr. yellow crystals: mp 25−30 °C. ^{1}H NMR ($CDCl_3$): δ 1.63 (t, J = 7.7 Hz, 3H, CH_3), 2.92 (q, J = 7.7 Hz, 2H, CH_2Te), 7.25−7.56 (m, 4H, Ar). ^{13}C NMR ($CDCl_3$): δ 4.1 (CH_2Te), 17.6 (CH_3), 128.0, 129.0, 139.8, 141.4 (Ar), 194.7 (C═O). ^{125}Te NMR (CD_3OD): δ 801.7, MS (CI): *m*/*z* 299, 297, 295 (M + 1). Anal. Calcd for C_9H_9ClOTe: C, 36.49; H, 3.06. Found: C, 34.12; H, 3.11.

***Te*-Methyl 3-Chloro-2,6-dimethoxybenzenecarbotelluroate (3zl). Method A using sodium salt 1t:** yellow oil (92%). ^{1}H NMR ($CDCl_3$): δ 2.15 (s, 3H, CH_3Te), 3.75 (s, 3H, CH_3O), 3.80 (s, 3H, CH_3O), 6.56−7.29 (m, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ −13.1 (CH_3-Te), 56.1 (CH_3O), 62.5 (CH_3O), 108.3, 120.1, 130.1, 132.1, 151.7, 154.6 (Ar), 191.1 (C═O). MS (CI): *m*/*z* 347, 346, 345, 343, 341, 340 (M + 1), 199 (3-Cl-2,6-$(CH_3O)_2C_6H_2CO^+$).

***Te*-Ethyl 3-Chloro-2,6-dimethoxybenzenecarbotelluroate (3zm). Method A using sodium salt 1t:** yellow oil (86%). ^{1}H NMR ($CDCl_3$): δ 1.67 (t, J = 7.7 Hz, 3H, CH_3), 2.89 (q, J = 7.7 Hz, 2H, CH_2Te), 3.73 (s, 3H, CH_3O), 3.79 (s, 3H, CH_3O), 6.54−7.26 (m, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ 4.2 ($TeCH_2$), 17.5 (CH_3), 56.0 (CH_3O), 62.4 (CH_3O), 108.2, 119.8, 130.6, 131.8, 151.2, 154.2 (Ar), 192.0 (C═O). MS (CI): *m*/*z* 359, 357, 355, 354, 353 (M + 1), 199 (3-Cl-2,6-$(CH_3O)_2C_6H_2CO^+$).

***Te*-2-Methylpropyl 3-Chloro-2,6-dimethoxybenzenecarbotelluroate (3zn). Method A using sodium salt 1t:** yellow oil (57%). ^{1}H NMR ($CDCl_3$): δ 0.94 (d, J = 6.6 Hz, 6H, CH_3), 1.91 (m, J = 6.6 Hz, 1H, CH), 2.95 (d, J = 6.6 Hz, 2H, CH_2), 3.74 (s, 3H, CH_3O), 3.79 (s, 3H, CH_3O), 6.54−7.27 (m, 2H, Ar). ^{13}C NMR ($CDCl_3$): δ 23.7 ($TeCH_2$), 24.1 (CH), 29.9 (CH_3), 56.2 (CH_3O), 62.5 (CH_3O), 108.3, 120.0, 131.1, 131.8, 151.2, 154.1 (Ar), 192.0 (C═O). ^{125}Te NMR ($CDCl_3$): δ 410.2. HRMS = $C_{12}H_{16}O_2Te$, calcd 322.0212 (^{130}Te); found 322.0222 (^{130}Te).

***Te*-Methyl 1-Naphthalenecarbotelluroate (3zo). Method A using sodium salt 1u:** yellow crystals (77%): mp: 26−28 °C. ^{1}H NMR ($CDCl_3$): δ 2.12 (s, 3H, CH_3Te), 7.24−8.47 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$): δ −12.1 (CH_3Te), 124.5, 124.9, 126.3, 126.7, 127.8, 128.2, 128.7, 133.0, 133.6, 140.5 (Ar), 198.2 (C═O); MS (CI): *m*/*z* 301, 299, 297, 296, 295 (M + 1), 155 (1-$C_{10}H_7CO^+$).

***Te*-Ethyl 1-Naphthalenecarbotelluroate (3zp). Method A using sodium salt 1u:** yellow oil (75%). ^{1}H NMR ($CDCl_3$): δ 1.80 (t, J = 7.7 Hz, 3H, CH_3), 3.05 (q, J = 7.7 Hz, 2H, CH_2Te), 7.43−8.63 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$): δ 5.3 (CH_2Te), 17.7 (CH_3), 124.6, 125.1, 126.5, 126.8, 128.0. 128.4, 129.0, 133.1, 133.8, 141.0 (Ar), 199.1 (C═O); MS (CI): *m*/*z* 315, 313, 311, 309 (M + 1), 155 (1-$C_{10}H_7CO^+$).

***Te*-Ethyl 2-Naphthalenecarbotelluroate (3zq). Method A using sodium salt 1v:** yellow crystal (62%). Mp: 61−63 °C dec. ^{1}H NMR ($CDCl_3$): δ 1.57 (t, J = 7.7 Hz, 3H, CH_3), 3.05 (q, J = 7.7 Hz, 2H, CH_2Te), 7.52−8.25 (m, 10H, Ar). ^{13}C NMR ($CDCl_3$): δ 3.9 (CH_2Te), 17.8 (CH_3), 122.4, 127.1, 127.9, 128.6, 128.8, 129.0, 129.6, 132.6, 136.0, 140.5 (Ar), 196.1 (C═O). MS (CI): *m*/*z* 315, 313, 311 (M + 1), 155 (2-$C_{10}H_7CO^+$).

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Supporting Information Available: Complete listings of bond distances, angles and torsion angles, thermal parameters, and atomic coordinates for compound **3zj**. This material is available free of charge via the Internet at http://pubs.acs.org. Ordering information is given on any current masthead page.

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