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**Synthesis and application of trimethylsilyl dinitramide
Me₃SiN(NO₂)₂ as dinitramide transfer reagent;
preparation of covalent organotellurium(IV)
dinitramides**

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**Synthesis and application of trimethylsilyl dinitramide
Me₃SiN(NO₂)₂ as dinitramide transfer reagent; preparation
of covalent organotellurium(IV) dinitramides**

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17 **Synthesis and application of trimethylsilyl dinitramide $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ as**
18 **dinitramide transfer reagent; preparation of covalent organotellurium(IV)**
19 **dinitramides**
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37 Dedicated to Prof. Dr. Gerd Meyer, on the occasion of his 60th birthday
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41 Received
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Abstract

The preparation and properties of trimethylsilyl dinitramide $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ are reported. Its application as dinitramide transfer reagent furnished the first covalent organotellurium(IV) bis(dinitramides) $\text{R}_2\text{Te}[\text{N}(\text{NO}_2)_2]_2$, which were characterized by multinuclear NMR spectroscopy. Alternatively, $\text{R}_2\text{Te}[\text{N}(\text{NO}_2)_2]_2$ were synthesized from the corresponding organotellurium(IV) halides R_2TeX_2 with silver dinitramide.

Keywords: Trimethylsilyl dinitramide, Tellurium; Dinitramides; Multinuclear NMR spectroscopy

Introduction

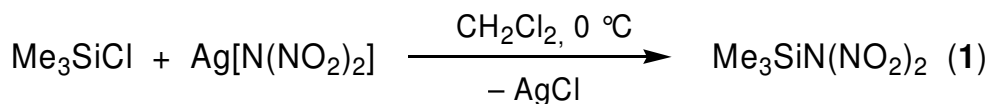
As an alternative azide transfer reagent, trimethylsilyl azide Me_3SiN_3 , has wide application possibilities and some advantages compared to silver azide.[1] Similarly, for example Me_3SiCN and Me_3SiCF_3 , have been proven to be excellent reagents for transferring a cyanide- and trifluoromethyl-group respectively.[2-9] In general the use of the respective silver salts usually necessitate chlorides, bromides or iodides as starting materials. The separation of the resulting precipitate AgX ($\text{X} = \text{Cl}, \text{Br}, \text{I}$) is especially in case of temperature-, moisture- or air-sensitive materials a considerable problem. In addition the light-sensitivity of silver salts, and, especially in the case of AgN_3 , the extreme sensitivity towards shock and friction comprises some risks and avoids straightforward syntheses. The reactions with commercially available Me_3SiN_3 , which is less hazardous compared to AgN_3 , result only in the formation of Me_3SiF as by-product, which can easily be removed in vacuo. Therefore, the preparation of the analogous compound $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ and application as dinitramide transfer reagent would be of considerable interest.

Not at least because of our research in the past years, the chemistry of tellurium halides as precursors for the corresponding tellurium(IV) azides is well investigated.[10-17] Recently, we reported the successful preparation and characterization of organochalcogenium dinitramides.[18] In this contribution, as a consequence, we also focus on the preparation of the first covalent organotellurium(IV) dinitramides.

Results and Discussion

The preparation of trimethylsilyl dinitramide $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ (**1**) was achieved by reaction of Me_3SiCl with silver dinitramide in dichloromethane as solvent (Scheme 1). Immediately after the addition of silver dinitramide the formation of AgCl as precipitate was

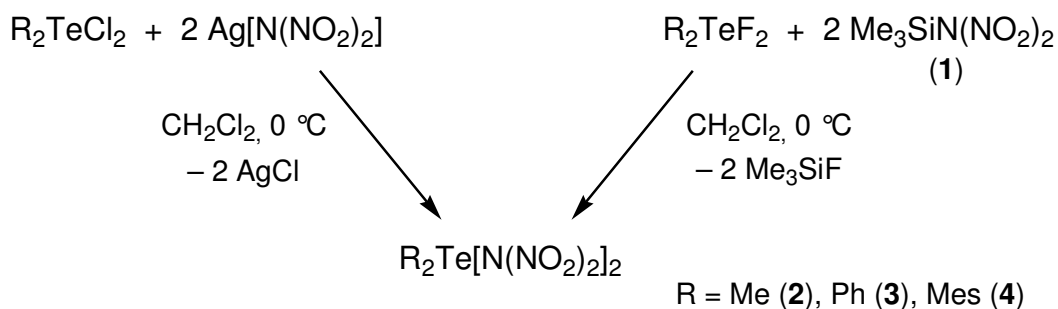
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3 obtained. After stirring for one hour at 0 °C, the NMR spectra of the solution indicate the
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5 formation of **1**.
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14 Scheme 1: Preparation of Me₃SiN(NO₂)₂ (**1**).
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19 The pale yellow solution displays two resonances in the ¹⁴N NMR spectrum at δ = -24
20 and -101 ppm (solvent CH₂Cl₂). In contrast to silver dinitramide,[19] the resonance of the
21 amide nitrogen in **1** is significantly shifted to higher field. In the ²⁹Si NMR spectrum
22 (δ = 6.8 ppm) the resonance is in-between the resonances for Me₃SiCN (δ = -12.3 ppm) and
23 Me₃SiN₃ (δ = 15.9 ppm),[20] and considerably different compared to the resonance of the
24 starting material Me₃SiCl (δ = 30.2 ppm).[20] The silyl dinitramide **1** is storable in solution
25 for at least six hours. Evaporation of the solvent at 0 °C resulted in the formation of a
26 brownish, viscous residue, which could not be dissolved again. Therefore an isolation of neat
27 **1** was not possible. However, the application as dinitramide transfer reagent for the
28 preparation of the first covalent organotellurium(IV) dinitramides was successful.
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46 For the preparation of diorganotellurium(IV) bis(dinitramides) two different reaction
47 pathways have been investigated (Scheme 2). Starting from the corresponding dichlorides
48 R₂TeCl₂, the reaction with silver dinitramide or its pyridine or acetonitrile solvates[19, 21]
49 resulted in the formation of the corresponding organotellurium(IV) dinitramides
50 R₂Te[N(NO₂)₂]₂ (R = Me (**2**), Ph (**3**), Mes (**4**)).
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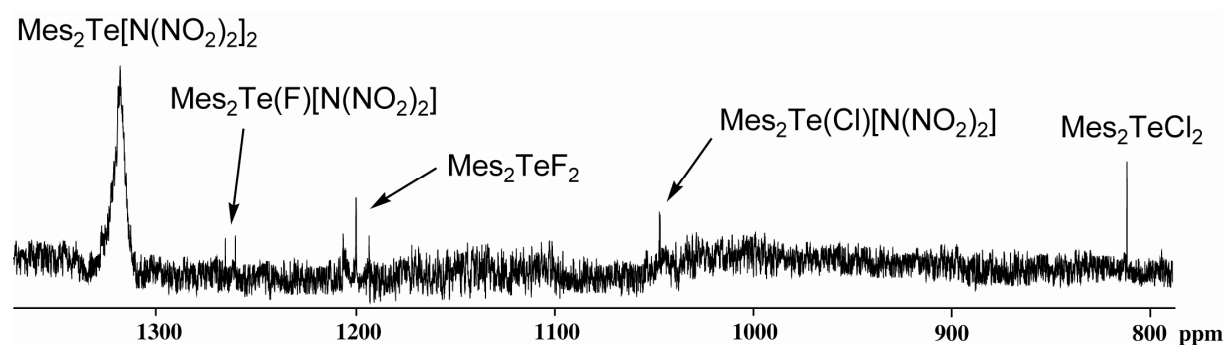


Scheme 2: Preparation of covalent organotellurium(IV) bis(dinitramides)



The second reaction pathway with the help of the new trimethylsilyl dinitramide (**1**) requires the corresponding organotellurium(IV) difluorides R_2TeF_2 as starting materials. The reaction was performed with a freshly prepared solution of **1** in CH_2Cl_2 . According to the ^{125}Te NMR spectra both pathways resulted in the same products, which unambiguously proves that **1** can be used as *in situ* generated dinitramide transfer reagent. In the ^{19}F NMR spectra of the reaction mixtures, the resonance of Me_3SiF ($\delta = -158.4$ ppm) was observed as the major resonance. The ^{125}Te NMR resonances of the dinitramides **2**, **3** and **4** in CDCl_3 (see Table 1) are shifted to lower field compared to the resonances of the corresponding difluorides (Me_2TeF_2 : $\delta = 1232$ ppm, Ph_2TeF_2 : $\delta = 1128$ ppm, Mes_2TeF_2 : $\delta = 1206$ ppm).[22] In the ^{14}N NMR spectra the resonance of the nitro group is observed in the range between $\delta = -16$ and -20 ppm, and comparable to ionic triorganochalcogenium dinitramide $[\text{R}_3\text{Ch}][\text{N}(\text{NO}_2)_2]$ ($\text{Ch} = \text{Te, Se, S}$; $\text{R} = \text{Ph, Me}$) salts ($\delta = -11$ to -14 ppm).[18] In accordance with the covalent silyl derivative **1**, the resonances of the amide nitrogen atoms are significantly shifted to higher field (see Table 1) compared to the ionic compounds ($\delta = -55$ to -59 ppm).[18] In contrast to the resonances of the nitro groups, the amide resonances of **2–4** are significantly broadened with line widths larger than 550 Hz.

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3 If a solution of Mes_2TeF_2 in CH_2Cl_2 is reacted with a substoichiometrical amount of an
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5 *in situ* prepared solution of $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ (**1**) in CH_2Cl_2 , the resonance of the tellurium(IV)
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7 bisdinitramide **4** ($\delta = 1334$ ppm) is found to be the major product (Figure 1). Furthermore,
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9 apart from the resonance of the starting material Mes_2TeF_2 ($\delta = 1205$ (t) ppm), also
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11 resonances for $\text{Mes}_2\text{TeCl}_2$ ($\delta = 813$ ppm), resulting from reaction with still present Me_3SiCl ,
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13 and likely the two mixed species $\text{Mes}_2\text{Te}(\text{F})[\text{N}(\text{NO}_2)_2]$ ($\delta = 1269$ (d) ppm) and
14
15 $\text{Mes}_2\text{Te}(\text{Cl})[\text{N}(\text{NO}_2)_2]$ ($\delta = 1048$ ppm) as minor products are observed.
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35 Fig. 1: ^{125}Te NMR spectrum of the reaction of Mes_2TeF_2 with a
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37 substoichiometrical amount of *in situ* prepared $\text{Me}_3\text{SiN}(\text{NO}_2)_2$ (**1**) (very diluted
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39 solution in CH_2Cl_2).
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44 The IR and Raman spectra of the compounds **3** and **4** display, as expected, similar
45
46 bands compared to the ionic organochalcogenium dinitramides (for details see Experimental
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48 Section).
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51 Whereas the methyl substituted derivative **2** decomposes within hours, the phenyl and
52
53 mesityl substituted compounds are considerably more stable. According to ^{125}Te NMR
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55 spectroscopy, the storage of **3** and **4** at ambient temperature under an inert atmosphere of dry
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57 argon did not indicate any signs of decomposition after two weeks. However, after a period of
58
59 two months both compounds also showed decomposition to unidentifiable products, likely
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3 oxygen containing materials such as the corresponding diorganotellurium(IV) oxides R_2TeO .
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5 Besides nitric acid, N_2O could unambiguously be identified in ^{14}N NMR spectra ($\delta = -148, -$
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7
8 232 ppm in $CDCl_3$) as further decomposition products of the organotellurium(IV)
9
10 bis(dinitramides).

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15 During the preparation as well as during handling and characterization attention
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17 should be paid to the fact that the stability of covalent dinitramides differs from the stability
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19 of ionic dinitramides. Covalent dinitramides are less stable than the ionic dinitramides, which
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21 is in accordance with stability differences between the corresponding covalent and ionic
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23 nitrates and nitrites respectively.[23] The reason for instability of a covalent dinitramide
24
25 moiety likely is the result of a sterical hindering of the two nitro-groups, which is significantly
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27 different to an ionic dinitramide, and the comparatively high electronegativity of the *N,N*-
28
29 dinitro-group. Furthermore the absence of an additional electron, which accounts in the
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31 dinitramide-anion for resonance stabilization and thereby resulting in a higher N–N bond
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33 order, decreases the stability.[24] Therefore the few up to now reported alkyldinitramines
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35 decompose at temperatures below 70 °C and are extremely shock sensitive.[23]
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41 In summary the first covalent organotellurium(IV) bis(dinitramides) were synthesized
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43 by reaction of the organotellurium(IV) dihalides with silver dinitramide or with trimethylsilyl
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45 dinitramide as a new dinitramide transfer reagent. The silyl derivative could also be
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47 characterized by multinuclear NMR spectroscopy and its limited stability was noted.
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51 52 53 54 55 **Experimental Section**

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58 *General remarks*
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3 All manipulation of air- and moisture-sensitive materials were performed under an
4 inert atmosphere of dry argon using flame-dried glass vessels or oven-dried plastic equipment
5 and Schlenk techniques;^[25] solvents were freshly distilled and stored under N₂. Silver
6 dinitramide reagents^[19, 21] and organotellurium(IV) dihalogenides R₂TeX₂ (R = Me, Ph,
7 Mes; X = F, Cl)^[26] were prepared according to literature procedures. Me₃SiCl was freshly
8 distilled prior to use. Infrared spectra were recorded on Perkin-Elmer Spektrum One FT-IR or
9 Nicolet 520 FT-IR spectrometers (as KBr pellets or between KBr plates), Raman spectra on
10 Perkin-Elmer 2000 NIR FT spectrometer fitted with a Nd:YAG laser (1064 nm, 200 mW).
11 NMR spectra were recorded on a JEOL Eclipse 400 instrument at 25 °C, and chemical shifts
12 were determined with respect to external Me₄Si (¹H, 399.8 MHz; ¹³C, 100.5 MHz; ²⁹Si,
13 79.5 MHz), MeNO₂ (¹⁴N, 28.9 MHz), and Me₂Te (¹²⁵Te, 126.1 MHz). Due to the high
14 sensitivity of the compounds, elemental analyses could not be performed satisfactory and are
15 therefore not reported. The preparation of covalent organotellurium(IV) bisdinitramides
16 afforded products with yields in the range between 60 and 85 %.

37 38 *Experimental procedures and analytical data*

39 40 **Me₃SiN(NO₂)₂ (1)**

41
42 Into a solution of 2.1 mmol Me₃SiCl in 5 mL CH₂Cl₂ were added 2.1 mmol Ag[N(NO₂)₂] at
43 0 °C. After stirring the solution for 1 h at 0 °C, a sample for NMR experiments was prepared.
44
45 Evaporation of all volatile materials resulted in the formation of nitrous gases and a remaining
46 brownish solid, which could not be identified.

47
48 ¹H NMR (CH₂Cl₂/CDCl₃): 0.26 (²J_{H-29Si} = 6.9 Hz) ppm; ¹³C{¹H} NMR (CH₂Cl₂/CDCl₃): -0.9
49 (¹J_{C-29Si} = 58.5 Hz) ppm; ¹⁴N NMR (CH₂Cl₂/CDCl₃): -24 (N(NO₂)₂), -101 (br, N(NO₂)₂)
50 ppm; ²⁹Si{¹H} NMR (CH₂Cl₂/CDCl₃): 6.8 ppm.
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3 *General procedure for the preparation of the diorganotellurium(IV) bis(dinitramides)*
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6 $R_2Te[N(NO_2)_2]_2$

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8 *Method I:* Into a solution of 0.78 mmol R_2TeCl_2 in 10 mL CH_2Cl_2 were added 1.56 mmol
9
10 $Ag[N(NO_2)_2]$ at 0 °C. After stirring for 2 h at 0 °C, the colorless precipitate (AgCl) was
11
12 separated and all volatile materials removed in vacuo. The bis(dinitramides) **2–4** were
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14 obtained as pale-yellow oils, which decompose according to ^{125}Te NMR spectra within a few
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16 hours (**2**) or a couple of weeks (**3** and **4**), respectively.
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20 *Method II:* Into a solution of 0.65 mmol R_2TeF_2 in 5 mL CH_2Cl_2 were added 1.30 mmol of *in*
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22 *situ* prepared **1** in CH_2Cl_2 at 0 °C. NMR experiments of the solutions and of isolated products
23
24 furnish the same NMR resonances according to *Method I*.
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29 **$Me_2Te[N(NO_2)_2]_2$ (**2**)**

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31 1H NMR ($CDCl_3$): 1.73 ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): 18.6 ppm; ^{14}N NMR ($CDCl_3$): -20
32
33 ($N(\underline{NO}_2)_2$), -104 (br, $\underline{N}(\underline{NO}_2)_2$) ppm; $^{125}Te\{^1H\}$ NMR ($CDCl_3$): 1295 ppm.
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39 **$Ph_2Te[N(NO_2)_2]_2$ (**3**)**

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41 IR (KBr): 3416 br, 3053 m, 2946 m, 1631 m, 1604 m, 1585 m, 1528 vs, 1501 w, 1474 s, 1462
42
43 m, 1381 s, 1332 m, 1291 w, 1258 m, 1144 vs, 1006 s, 853 m, 820 w, 742 s, 688 m, 678 m,
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45 614 s, 535 $m\text{ cm}^{-1}$. Raman: 3002 (45), 1598 (56), 1582 (42), 1510 (48), 1420 (32), 1395 (40),
46
47 1300 (81), 1284 (70), 1253 (65), 1199 (55), 1004 (100), 825 (45), 678 (49), 615 (65), 558
48
49 (70), 471 (55), 456 (25), 411 (48), 341 (56), 297 (50), 228 (51), 170 (59) cm^{-1} .
50
51

52
53 1H NMR ($CDCl_3$): 7.90/7.58 (m, Ar-H) ppm; $^{13}C\{^1H\}$ NMR ($CDCl_3$): 132.5 (*o*-C), 131.1 (*p*-
54
55 C), 130.6 (*m*-C), 126.4 (C-Te, $^1J_{C-125Te} = 265.1$ Hz) ppm; ^{14}N NMR ($CDCl_3$): -18 ($N(\underline{NO}_2)_2$),
56
57 -105 (br, $\underline{N}(\underline{NO}_2)_2$) ppm; $^{125}Te\{^1H\}$ NMR ($CDCl_3$): 1286 ppm.
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Mes₂Te[N(NO₂)₂]₂ (4)

IR (KBr): 3426 br, 3073 m, 2956 m, 1636 m, 1595 m, 1530 vs, 1484 s, 1451 s, 1384 s, 1342 w, 1292 w, 1184 vs, 1008 s, 853 w, 821 w, 751 s, 698 m, 680 s, 608 m, 539 m cm⁻¹. Raman: 3111 (86), 3022 (58), 2924 (86), 1635 (47), 1596 (62), 1553 (48), 1458 (50), 1383 (53), 1318 (72), 1295 (70), 1198 (56), 1012 (100), 825 (55), 688 (45), 609 (49), 554 (69), 474 (56), 412 (52), 347 (50), 296 (52), 228 (57), 176 (54) cm⁻¹.

¹H NMR (CDCl₃): 7.12/6.89 (m, *m*-CH, 4H), 2.91/2.53 (s, *o*-CCH₃, 12H), 2.30 (s, *p*-CCH₃, 6H) ppm; ¹³C{¹H} NMR (CDCl₃): 145.8/142.8 (*o*-C), 139.8 (*p*-C), 131.7/130.4 (*m*-C), 125.2 (C-Te), 24.6/24.1 (*o*-CH₃), 20.8 (*p*-CH₃) ppm; ¹⁴N NMR (CDCl₃): -16 (N(NO₂)₂), -101 (br, N(NO₂)₂) ppm; ¹²⁵Te{¹H} NMR (CDCl₃): 1334 ppm.

Table 1. ¹²⁵Te and ¹⁴N NMR resonances of the diorganotellurium(IV) bis(dinitramides) **2–4** (δ in ppm, solvent CDCl₃).

	$\delta^{125}\text{Te}$	$\delta^{14}\text{N}$ [N(<u>N</u> O ₂) ₂ / <u>N</u> (NO ₂) ₂]
Me ₂ Te[N(NO ₂) ₂] ₂ (2)	1295	-20/-104(br)
Ph ₂ Te[N(NO ₂) ₂] ₂ (3)	1286	-18/-105(br)
Mes ₂ Te[N(NO ₂) ₂] ₂ (4)	1334	-16/-101(br)

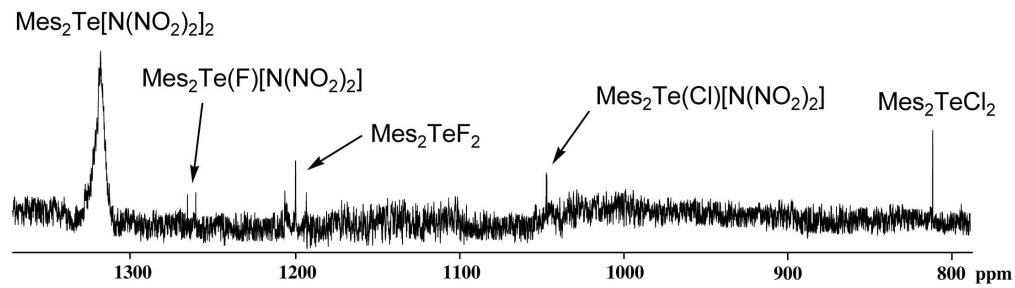
Acknowledgements

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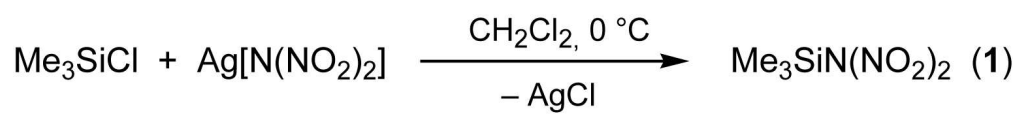
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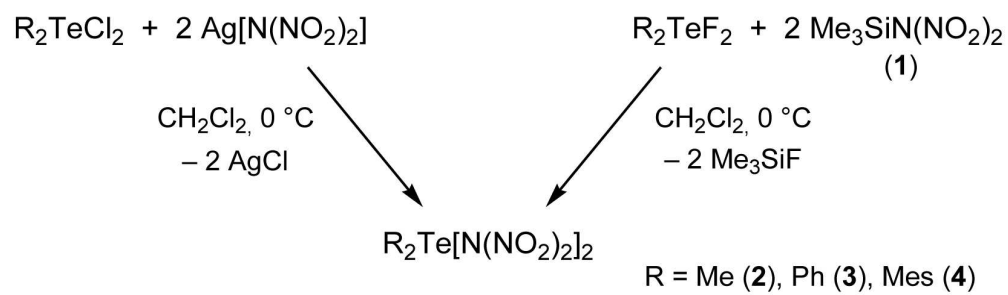
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185x51mm (300 x 300 DPI)



129x13mm (300 x 300 DPI)



140x40mm (300 x 300 DPI)