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Synthetic Applications of Alkyl (E)-2-Tributylstannyl-2-alkenoates: Selective Synthesis of (S)-1-Methylbutyl (E)-2-Methyl-2-pentenoate, an Aggregation Pheromone Component of Rhyzopertha dominica and Prostephanus truncatus

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SYNTHETIC APPLICATIONS OF ALKYL (E)-2-TRIBUTYLSTANNYL-2-ALKENOATES: SELECTIVE SYNTHESIS OF (S)-1-METHYLBUTYL (E)-2-METHYL-2-PENTENOATE, AN AGGREGATION PHEROMONE COMPONENT OF *Rhyzopertha dominica* AND *Prostephanus truncatus* [1)]

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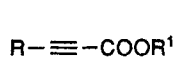
Abstract: Stereoisomerically pure (S)-1-methylbutyl (E)-2-methyl-2-pentenoate (*dominicalure-1*), (S)(E)-**11**, an aggregation pheromone component for the lesser and the greater grain borers, has been efficiently synthesized from the main product of the palladium-catalyzed reaction between ethyl 2-pentynoate, **1a**, and Bu_3SnH, *i.e.* ethyl (E)-2-tributylstannyl-2-pentenoate, (E)-**2a**.

Recently, we reported that the reaction between alkyl 2-alkynoates, **1**, and Bu_3SnH in THF solution at room temperature, in the presence of 2 mol % of $Pd(PPh_3)_4$, affords alkyl (E)-2-tributylstannyl-2-alkenoates, (E)-**2**, together with small amounts (2 – 9 %) of the corresponding alkyl (E)-3-tributylstannyl-2-alkenoates, (E)-**3**, in quite good yield[2].



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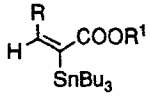
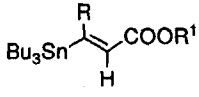


(E)-**2**



(E)-**3**

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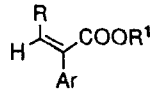
Moreover, we showed that regio- and stereoisomerically pure compounds (E)-**2**, which could be separated from these mixtures by MPLC on silica gel, represent effective precursors to stereodefined 2-(hetero)aryl substituted alkyl 2-alkenoates of general formula **4** as well as alkyl (E)-2-methyl-2-alkenoates, (E)-**5**, having very high stereoisomeric purity[2].



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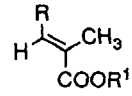
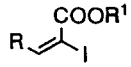
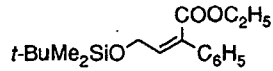


(E)-**5**

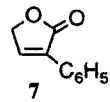
In particular, compounds **4** were efficiently synthesized by a simple reaction sequence which involved treatment of compounds (E)-**2** with an equimolar amount of iodine in CH_2Cl_2 solution, followed by a palladium-catalyzed reaction between (hetero)arylzinc chlorides and the iodo derivatives, (E)-**6**, so obtained[2]. Interestingly, one of these esters of general formula **4**, *i.e.* ethyl (Z)-4-(*tert*-butyldimethylsilyloxy)-2-phenyl-2-butenoate, (Z)-**4a**, has been recently used as direct precursor to 3-phenyl-5(H)-2-furanone, **7**[3], a metabolite of the hypnotic drug *glutethimide*, which can cause central nervous system depression when administered in large doses[4].



(E)-**6**

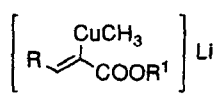


(Z)-**4a**

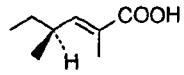


7

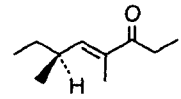
On the other hand, we found that treatment of compounds (E)-**6** with 3.5 equiv of $(CH_3)_2CuLi$ in Et_2O at -78 °C produced alkyl (Z)-(α-carbalkoxyvinyl)cuprate reagents of general formula (Z)-**8**, which were able to react with methyl iodide in HMPA – Et_2O to give stereoisomerically pure compounds (E)-**5** in quite good yields[2]. Noteworthy is that this procedure, which involves a configurational inversion, was employed to prepare the (S)-enantiomer of (E)-2,4-dimethyl-2-hexenoic acid , (S)(E)-**9**[2], a caste-specific substance of male carpenter ants in the genus *Camponotus*[5] and, more recently[3], 98% optically pure (S)(E)-4,6-dimethyl-4-octen-3-one, also named *manicone*, (S)(E)-**10**, an alarm pheromone component of ants in the genus *Manica*[6].



(Z)-8

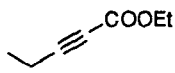


(S)(E)-9

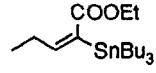


(S)(E)-10

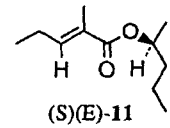
In continuation of the studies on the synthetic applications of alkyl (E)-2-tributylstannyl-2-alkenoates, (E)-**2**, we now describe an efficient synthesis of stereoisomerically pure (S)-1-methylbutyl (E)-2-methyl-2-pentenoate, (S)(E)-**11**, from the main product of the palladium-catalyzed reaction between ethyl 2-pentynoate, **1a**, and Bu_3SnH, *i.e.* ethyl (E)-2-tributylstannyl-2-pentenoate, (E)-**2a**[7]. Compound (S)(E)-**11**, also named *dominicalure-1*, is an aggregation pheromone component of the lesser grain borer, *Rhyzopertha dominica*[7a,8] as well as of the greater grain borer, *Prostephanus truncatus*[9] [Coleoptera, Bostrichidae]



1a



(E)-**2a**

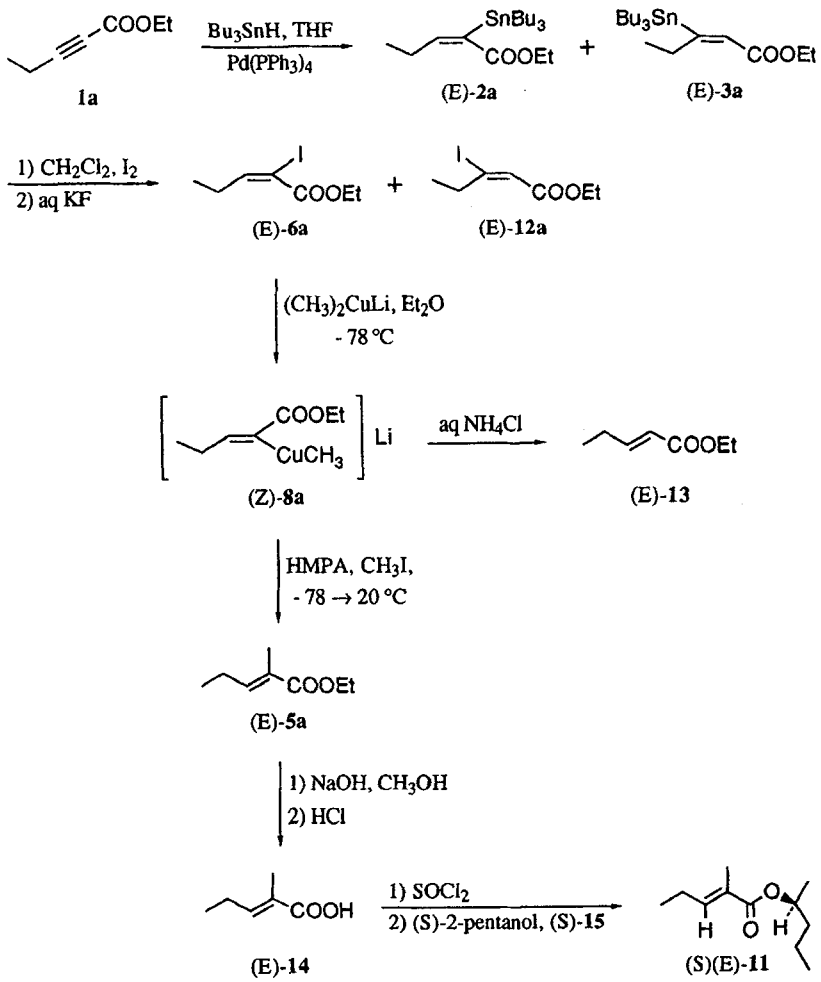


(S)(E)-**11**

The reaction sequence used to prepare compound (S)(E)-**11** is illustrated in the Scheme. Thus, compound **1a** was reacted with 0.97 equiv of Bu_3SnH in THF solution at room temperature, in the presence of *ca.* 2 mol % of $Pd(PPh_3)_4$, to give in 92% yield a mixture of ethyl (E)-2-tributylstannyl-2-pentenoate, (E)-**2a**, and ethyl (E)-3-tributylstannyl-2-pentenoate, (E)-**3a**, in a *ca.* 94:6 molar ratio, respectively. Since the separation on a multigram scale of compound (E)-**2a** from this mixture by MPLC on silica gel resulted to be quite difficult (see Experimental), this purified mixture was directly treated with an equimolar amount of iodine in CH_2Cl_2 solution at room temperature. The resultant reaction mixture, which contained ethyl (E)-2-iodo-2-pentenoate, (E)-**6a**, ethyl (E)-3-iodo-2-pentenoate, (E)-**12a** and Bu_3SnI was concentrated, diluted with Et_2O and, in order to eliminate Bu_3SnI, treated with a large excess of a semisaturated aqueous KF solution. The organic phase was filtered, concentrated and purified by MPLC on silica gel to give regio- and stereoisomerically pure (E)-**6a** in 79% yield based on

(E)-**2a**. Interestingly, concentration of the first eluted chromatographic fractions allowed to isolate a small amount of regioisomerically pure (E)-**12a**.

Scheme



Then, according to the procedure previously developed for the synthesis of alkyl (E)-2-methyl-2-alkenoates, (E)-**5**, starting from the corresponding alkyl (E)-2-iodo-2-alkenoates, (E)-**6**[2], compound (E)-**6a** was reacted with 3.6 equiv of $(CH_3)_2CuLi$ in Et_2O at -78 °C for 5 h. GLC/MS analysis of a sample of the reaction

mixture, which was hydrolyzed with aqueous NH_4Cl solution, showed the presence of ethyl (E)-2-pentenoate, (E)-**13**, together with a small amount of a compound subsequently identified as ethyl (E)-2-methyl-2-pentenoate, (E)-**5a**. This result suggested the presence of a (Z)-(α-carbalkoxyvinyl)cuprate reagent, *i.e.* (Z)-**8a**, in the reaction mixture. Therefore, this mixture was treated with HMPA at -78 °C, followed by addition of a molar excess of methyl iodide to give stereoisomerically pure ethyl (E)-2-methyl-2-pentenoate, (E)-**5a**, in 84% yield.

Saponification of this ester, followed by acidification gave in 85.2% yield (E)-2-methyl-2-pentenoic acid, (E)-**14**, which was converted to the corresponding acid chloride by treatment with $SOCl_2$ at 50 °C. Finally, this crude compound was reacted with a molar excess of commercially available enantiomerically pure (S)-2-pentanol, (S)-**15**, to give (S)-1-methylbutyl (E)-2-methyl-2-pentenoate, (S)(E)-**11**, in 58.8% yield. This ester had a value of the specific rotatory power, $[\alpha]^{25}_D$ +34.23 (c = 1.300, Et_2O), in satisfactory agreement with that reported for the naturally-occurring compound, $[\alpha]^{25}_D$ +32.1 (c = 0.156, Et_2O), which was assumed to be enantiomerically pure[7a]. However, this value was much higher than that more recently reported for a synthetic sample of (S)(E)-**11**, $[\alpha]_D$ +14.2 (c = 1.256, Et_2O)[7b].

EXPERIMENTAL

GLC analyses were performed on a Dani 6500 gas-chromatograph equipped with a Perkin Elmer LCI-100 integrator. Two types of capillary columns were used: a SRL-300 bonded FSOT column (30 m x 0.25 mm i.d.) and a SE-30 bonded FSOT column (30 m x 0.25 mm i.d.).

Purifications by MPLC were performed on a Büchi 681 instrument, using a Bischoff 8100 differential refractometer as detector.

GLC/MS analyses were performed using a VG 70/70E spectrometer interfaced with a Dani 3800 gas-chromatograph. ^1H NMR spectra were recorded on a Varian Gemini 200 MHz spectrometer using TMS as an internal standard. Optical rotations were measured on a Perkin-Elmer 142 polarimeter.

All reactions of air and water sensitive materials were performed in flame dried glassware under an atmosphere of argon or nitrogen. Air and water

sensitive solutions were transferred with hypodermic syringes or double-ended needles.

Ethyl 2-pentynoate, **1a** [b.p. 89-91 °C/37 Torr; lit[10] b.p. 78-80 °C/16-18 Torr] was prepared according to a general procedure[3,11] which involved treatment of the corresponding 1-alkyne, *i.e.* 1-butyne, with methyllithium in Et_2O – THF at -78 °C, followed by reaction of the resultant 1-lithium-1-alkyne with ethyl chloroformate. $Pd(PPh_3)_4$ was prepared according to the literature[12].

Ethyl (E)-2-tributylstannyl-2-pentenoate, (E)-2a

A degassed solution of Bu_3SnH (24.7 ml, 92.0 mmol) in dry THF (150 ml) was added during 2 h to a solution of ethyl 2-pentynoate, **1a** (12.0 g, 95.2 mmol) and $Pd(PPh_3)_4$ (2.29 g, 1.97 mmol) in THF (150 ml), which was stirred at room temperature under argon. After stirring for 3.5 h THF was removed under reduced pressure and the residue was diluted with hexane (1.2 l). After 1 h the precipitated palladium catalyst was removed by filtration on Celite and the filtrate was concentrated *in vacuo*. GLC/MS analysis of the residue showed the presence of two new compounds in a *ca.* 94:6 molar ratio. The first substance was subsequently identified as ethyl (E)-2-tributylstannyl-2-pentenoate, (E)-**2a**. The second compound could not be isolated but, on the basis of what previously reported on the palladium-catalyzed hydrostannation of alkyl 2-alkynoates[2,13], it was expected to correspond to ethyl (E)-3-tributylstannyl-2-pentenoate, (E)-**3a**. This compound had the following mass spectrum. MS, *m/z* (%): 361 (100), 360 (43), 359 (69), 358 (42), 357 (42), 305 (30), 249 (39), 247 (42), 179 (48), 177 (59), 175 (55), 165 (35), 163 (33), 127 (53), 121 (83), 119 (64), 99 (31).

The above mentioned residue was purified by MPLC on silica gel, using a mixture of hexane and Et_2O (99:1 *v/v*) as eluant. Concentration of the first eluted chromatographic fractions allowed to isolate a mixture of compounds (E)-**2a** and (E)-**3a** in a 91:9 molar ratio, respectively (24.76 g). On the other hand, concentration of the last eluted chromatographic fractions allowed to isolate chemically and stereoisomerically pure (E)-**2a** (10.53 g). ^1H NMR ($CDCl_3$): δ 6.04 (1H, t, J = 7.1 Hz, H-3), 4.15 (2H, q, J = 7.2 Hz, OCH_2), 2.43 (2H, *pseudo*-quint, J = 7.1 Hz, H-4), 1.70 – 1.20 (12H, m, H-2' and H-3'), 1.29 (3H, t, J = 7.2 Hz, O-C-CH_3), 1.15 – 0.75 ppm (18H, m, H-5, H-1' and H-4'). MS, *m/z* (%): 356 (16), 363 (16), 362

(16), 361 (100), 359 (76), 358 (36), 357 (46), 317 (22), 315 (23), 313 (15), 235 (14), 179 (34), 177 (32), 175 (23), 165 (31), 163 (22), 121 (21), 119 (16). Anal. Calcd. for $C_{19}H_{38}O_2Sn$: C, 54.70; H, 9.18. Found: C, 55.07; H, 9.34.

The chromatographic fractions which contained compounds (E)-**2a** and (E)-**3a** were then collected with those containing regio- and stereoisomerically pure (E)-**2a** to give a mixture of (E)-**2a** and (E)-**3a** (35.29 g, 92% yield based on Bu_3SnH) in a 94:6 molar ratio, respectively.

Ethyl (E)-2-iodo-2-pentenoate, (E)-6a

A solution of iodine (10.52 g, 41.42 mmol) in dry CH_2Cl_2 (470 ml) was added under argon during 3 h to a stirred CH_2Cl_2 solution (210 ml) of a mixture of compounds (E)-**2a** and (E)-**3a** (17.28 g, 41.42 mmol) in a 94:6 molar ratio, respectively. Upon completion of addition the reaction mixture was stirred for additional 2 h and concentrated *in vacuo*. The residue was diluted with Et_2O (350 ml) and stirred with a semisaturated aqueous KF solution (350 ml) at room temperature for 2.5 h. The reaction mixture was filtered and the filtrate was extracted with Et_2O. The organic extract was dried and concentrated *in vacuo*. GLC/MS analysis of the residue showed the presence of two compounds in a *ca.* 94:6 molar ratio, which were subsequently identified as ethyl (E)-2-iodo-2-pentenoate, (E)-**6a**, and ethyl (E)-3-iodo-2-pentenoate, (E)-**12a**, respectively. This residue was purified by MPLC on silica gel, using a mixture of hexane and Et_2O (99:1 *v/v*) as eluant. Concentration of the first eluted chromatographic fractions allowed to isolate compound (E)-**12a** (0.20 g). ^1H NMR ($CDCl_3$): δ 6.60 (1H, t, J = 0.7 Hz, H-2), 4.15 (2H, q, J = 7.1 Hz, OCH_2), 3.11 (2H, qd, J = 7.3 and 0.7 Hz, H-4), 1.28 (3H, t, J = 7.1 Hz, O-C-CH_3), 1.13 ppm (3H, t, J = 7.3 Hz, H-5). MS, *m/z* (%): 209 (M^+-OC_2H_5, 24), 128 (8), 127 (100), 99 (35), 82 (10), 81 (13), 71 (10), 55 (14), 54 (22), 53 (36).

On the other hand, concentration of the last eluted chromatographic fractions allowed to isolate regio- and stereoisomerically pure compound (E)-**6a** (7.78 g, 79% yield based on (E)-**2a**). ^1H NMR ($CDCl_3$): δ 6.89 (1H, t, J = 7.6 Hz, H-3), 4.25 (2H, q, J = 7.1 Hz, OCH_2), 2.47 (2H, *pseudo*-quint, J = 7.6 Hz, H-4), 1.33 (3H, t, J = 7.1 Hz, O-C-CH_3), 1.05 ppm (3H, t, J = 7.6 Hz, H-5). MS, *m/z* (%): 255 (M^+ +1, 8), 254 (M^+, 100), 226 (81), 225 (7), 209 (36), 208 (19), 181 (8), 127 (8), 99 (13), 98 (14), 81 (29), 71

(8), 55 (19), 54 (40), 53 (70), 43 (36). Anal. Calcd. for $C_7H_{11}IO_2$: C, 33.09; H, 4.36. Found: C, 32.78; H, 4.11.

Ethyl (E)-2-methyl-2-pentenoate, (E)-5a

A 1.73 M Et_2O solution of methyllithium (218.4 ml, 377.8 mmol) was slowly added to a suspension of CuI (35.87 g, 188.9 mmol) in Et_2O (320 ml) cooled to -78 °C. After stirring for 15 minutes a solution of ethyl (E)-2-iodo-2-pentenoate, (E)-**6a** (13.71 g, 53.09 mmol) in Et_2O (60 ml) was dropwise added and the reaction mixture was stirred for 5 h at -78 °C. GLC/MS analysis of a sample of this mixture, which was hydrolyzed with aqueous NH_4Cl, showed the presence of ethyl (E)-2-pentenoate, (E)-**13**, together with a small amount of a new compound subsequently identified as ethyl (E)-2-methyl-2-pentenoate, (E)-**5a**. HMPA (140 ml) and methyl iodide (50 ml, 803 mmol) were subsequently added and the resultant mixture was stirred for 0.5 h at -78 °C and for 14 h at room temperature. It was then poured into a large excess of a saturated aqueous NH_4Cl solution and extracted with Et_2O. The organic extract was filtered, washed with aqueous NH_4Cl, dried, concentrated *in vacuo* and fractionally distilled to give compound (E)-**5a** (6.45 g, 84% yield): b.p. 93-94 °C/52 Torr [lit[14] b.p. 66 °C/15 Torr (for a mixture of (E)- and (Z)-**5a**)]. ^1H NMR ($CDCl_3$): δ 6.75 (1H, tq, J = 7.5 and 1.4 Hz, H-3), 4.19 (2H, q, J = 7.1 Hz, OCH_2), 2.18 (2H, *pseudo*-dquint, J = 7.5 and 0.8 Hz, H-4), 1.83 (3H, dt, J = 1.4 and 0.8 Hz, CH_3-C=), 1.30 (3H, t, J = 7.1 Hz, O-C-CH_3), 1.05 ppm (3H, t, J = 7.5 Hz, H-5). MS, *m/z* (%): 142 (M^+, 45), 114 (25), 113 (23), 97 (74), 96 (12), 70 (11), 69 (100), 68 (11), 67 (14), 53 (11), 45 (11), 43 (17), 41 (92).

(E)-2-Methyl-2-pentenoic acid, (E)-14

A mixture of ethyl (E)-2-methyl-2-pentenoate, (E)-**5a** (6.40 g, 45.0 mmol), NaOH (20.5 g , 512 mmol) and methanol (200 ml) was stirred for 16 h at room temperature and for 1.5 h under reflux. It was then cooled to 20 °C and concentrated *in vacuo*. The residue was diluted with water (100 ml) and extracted with Et_2O. The aqueous layer was acidified with 10% HCl and extracted with Et_2O. The organic extract was washed with brine, dried, concentrated *in vacuo* and distilled to give compound (E)-**14** (4.38 g, 85.2% yield): b.p. 113 °C/13 Torr (lit [15] b.p. 123-125 °C/30 Torr). ^1H NMR ($CDCl_3$): δ 12.05 (1H, br s, COOH), 6.91 (1H, tq, J = 7.5 and 1.4 Hz, H-3), 2.20 (2H, *pseudo*-quint, J = 7.5 and 0.9 Hz, H-4), 1.83

(3H, dq, J = 1.4 and 0.9 Hz, CH_3-C=), 1.06 ppm (3H, t, J = 7.5 Hz, H-5). The spectral properties of this compound were in satisfactory agreement with those previously reported[15].

(S)-1-Methylbutyl (E)-2-methyl-2-pentenoate, (S)(E)-11

A mixture of compound (E)-**14** (0.845 g, 7.40 mmol) and thionyl chloride (1.10 g, 9.25 mmol) was heated to 50 °C for 20 minutes. After this period the excess of thionyl chloride was removed in vacuo (26 Torr) and the crude carboxylic acid chloride so obtained was treated with commercially available enantiomerically pure (S)-2-pentanol, (S)-**15** (0.75 g, 8.51 mmol), $[\alpha]^{25}_D$ + 13.7 ± 0.5 (neat) at 50 °C for 0.5 h. After this period the reaction mixture was cooled to room temperature, poured into an excess of a diluted aqueous $NaHCO_3$ solution and extracted with Et_2O. The organic extract was washed with water until neutrality, dried and concentratyed *in vacuo*. The residue was purified by MPLC on silica gel, using a mixture of hexane and Et_2O (99:1 *v/v*) as eluant, followed by distillation (bulb-to-bulb) to give compound (S)(E)-**11** (0.80 g, 58.8% yield): $[\alpha]^{25}_D$ +34.23 (c = 1.300, Et_2O) [lit[7a] $[\alpha]^{25}_D$ + 32.1 (c = 0.156, Et_2O)]. ^1H NMR ($CDCl_3$): δ 6.72 (1H, tq, J = 7.4 and 1.4 Hz, H-3), 4.97 (1H, *pseudo*-sext, J = 6.3 Hz, H-2'), 2.18 (2H, *pseudo*-quint, J = 7.4 Hz, H-4), 1.82 (3H, d, J = 1.4 Hz, CH_3-C=), 1.75 -1.15 (4H, m, H-3' and H-4'), 1.24 (3H, d, J = 6.3 Hz, H-1'), 1.05 (3H, t, J = 7.4 Hz, H-5), 0.92 ppm (3H, t, J = 7.1 Hz, H-5'). MS, *m/z* (%): 184 (M^+, 3), 155 (5), 116 (8), 115 (93), 114 (5), 98 (11), 97 (100), 87 (11), 71 (13), 70 (62), 69 (56), 55 (20), 43 (55), 42 (14), 41 (60)).

REFERENCES AND NOTES

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