# ORGANOTITANIUM CHEMISTRY

## V *. ELECTRON IMPACT AND NEGATIVE ION CHEMICAL IONIZATION MASS SPECTRA OF DERIVATIVES OF DICYCLOPENTADIENYLTITANIUM(IV) DICHLORIDE AND ALLYLDICYCLOPENTADIENYLTITANIUM(III)

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(Received February 24th, 1986; in revised form April 28th, 1986)

## Summary

Electron impact (EI) and negative ion chemical ionization (NCI) mass spectra of twenty-four derivatives of dicyclopentadienyltitanium(IV) dichloride and eleven derivatives of allyldicyclopentadienyltitanium(III) were investigated. For EI spectra of these 24 compounds most of the molecular ion peaks were not discernible. The characteristic fragment ions $(M - Cl)^+$, $(M - 2Cl)^+$, $(M - 2HCl)^+$, $(M - RC_5H_4)^+$ and $(RC_5H_4)^+$ were observed. The NCI mass spectra of these 24 compounds exhibited strong molecular ion peaks and a series of ions giving structural information.

The EI mass spectra of $\eta^3$-allyldi-$\eta^5$-cyclopentadienyl-titanium(III) compounds showed molecular ion peaks with low intensity and a series of cyclopentadienylitanium ions and ions characteristic of allyl groups.
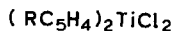
## Introduction

The mass spectra of $(\eta^5-C_5H_5)_2MX_2$ (where M = Ti, Zr, Hf; X = F, Cl, Br, I) [1–4] have been reported. The spectra are relatively simple and show competitive loss of the halide and $C_5H_5$ after ionization. In all cases, the highest $m/z$ ion is the monomeric parent molecular ion. In this paper, an extensive study has been made of twenty-four derivatives of dicyclopentadienyltitanium(IV) dichloride and eleven
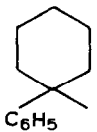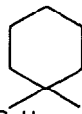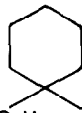
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* For part IV see ref. [8]

derivatives of allyldicyclopentadienyltitanium(III). The fragmentation pathways are discussed.

## Results and discussion

The electron impact (EI) mass spectra of compounds $(\eta^5\text{-RC}_5\text{H}_4)_2\text{TiCl}_2$, **1–24**, are

$$( RC_5H_4)_2TiCl_2$$

1. R = CH$_3$
2. R = C$_2$H$_5$
3. R = n-C$_3$H$_7$
4. R = n-C$_4$H$_9$
5. R = n-C$_5$H$_{11}$
6. R = CH$_3$(CH$_2$)$_2$ CH
   |
   CH$_3$
7. R = cyclo - C$_5$H$_9$
8. R = cyclo - C$_6$H$_{11}$

9. R = CH$_2$=CHCH$_2$
10. R = CH$_3$CH=CHCH$_2$
11. R = CH$_2$=CCH$_2$
    |
    CH$_3$
12. R = (CH$_3$)$_2$C=CHCH$_2$
13. R = CH$_2$=CHC(CH$_3$)$_2$
14. R = CH$_2$=CHCH
    |
    n-C$_3$H$_7$
15. R = CH$_2$=CHCH$_2$CH=CHCH$_2$
16. R = CH$_3$OCH$_2$CH$_2$

17. R = C$_2$H$_5$OCH$_2$CH$_2$

18. R =
C$_6$H$_5$

19. R =
m-CH$_3$C$_6$H$_4$

20. R =
p-CH$_3$C$_6$H$_4$

21. R = C$_6$H$_5$C(CH$_3$)$_2$
22. R = C$_6$H$_5$C(CH$_3$)(C$_2$H$_5$)
23. R = C$_6$H$_5$C(C$_2$H$_5$)$_2$
24. R = p-CH$_3$OC$_6$H$_4$C(CH$_3$)$_2$

listed in Table 1 (only ions of importance are listed). In most of these spectra, molecular ion peaks were not discernible; only for compounds with an alkyl substituent were molecular ion peaks of low intensity observed. The major degradation pathway of the molecular ions formed from the complexes involved the successive elimination of chlorine to form intense peaks corresponding to $(M - \text{Cl})^+$ and $(M - 2\text{Cl})^+$, respectively. In addition to the elimination of two chlorines; elimination of 2HCl also occurred competitively. Another degradation pathway consisted of elimination of the substituted cyclopentadienyl ligand giving intense peaks corresponding to the $(M - \text{RC}_5\text{H}_4)^+$ ion. Elimination of $\text{C}_2\text{H}_6$, $\text{C}_3\text{H}_6$ and 2H from n-C$_4$H$_9$C$_5$H$_4$ produced intense peaks at $m/z$ 91, 79 and 77, respectively (Scheme 1).

Unlike the spectra of derivatives of unsubstituted cyclopentadienyl, the spectra of compounds **1–8** contain a very intense peak corresponding to $(M - 2\text{HCl})^+$, which has a greater intensity than that of $(M - 2\text{Cl})^+$. Introduction of an alkenyl group or another complex substituent into the cyclopentadienyl ring (**9–24**) causes an increase in the intensity of the $(M - 2\text{Cl})$ peak which is greater that of the $(M - 2\text{HCl})$ peak (excepting compound **15**) (see Table 1). Of special interest is the occurrence of a prominent adduct ion $(M - \text{Cl} + \text{C}_n\text{H}_{2n-2})$ $(n = 3\text{–}4)$ in the spectra of compounds **9–11**. The product was postulated to have arisen from the reaction of the initial $(M - \text{Cl})^+$ product with a neutral molecule $\text{C}_n\text{H}_{2n-2}$ $(n = 3\text{–}4)$.

$(C_7H_7)^+$ $\xleftarrow{-C_2H_6}$ $(n\text{-}C_4H_9C_5H_4)^+$ $\xrightarrow{-C_3H_6}$ $(C_6H_7)^+$ $\xrightarrow{-2H}$ $(C_6H_5)^+$

$m/z$ 91       $m/z$ 121       $m/z$ 79       $m/z$ 77

$(n\text{-}C_4H_9C_5H_4TiCl_2)^+$ $\xleftarrow{-n\text{-}C_4H_9C_5H_4}$ $((n\text{-}C_4H_9C_5H_4)_2TiCl_2)^{+\cdot}$ $\xrightarrow{-\cdot Cl}$ $(M-Cl)^+$ $\xrightarrow{-\cdot Cl}$ $(M-2Cl)^{+\cdot}$

$m/z$ 239       $M^{+\cdot}, m/z$ 360       $m/z$ 325       $m/z$ 290

$\downarrow -HCl$       $\downarrow -2HCl$       $\downarrow -C_4H_8$       $\downarrow -C_4H_8$

$(C_9H_{12}TiCl)^+$       $(M-2HCl)^{+\cdot}$       $(M-Cl-C_4H_8)^+$       $(M-2Cl-C_4H_8)^{+\cdot}$

$m/z$ 203       $m/z$ 288       $m/z$ 269       $m/z$ 234

$\downarrow -C_4H_8$

$(M-2HCl-C_4H_8)^{+\cdot}$

$m/z$ 232

**SCHEME 1**



SCHEME 2. Possible fragmentation pathways for compounds 25–35.

TABLE 1

EI MASS SPECTRAL DATA OF COMPOUNDS **1–24** (relative intensities in parentheses)

| Compound | $M^+$ | $(M-Cl)^+$ | $(M-2Cl)^+$ | $(M-2HCl)^+$ | $(M-RC_5H_4)^+$ | $M\genfrac{}{}{0pt}{}{-RC_5H_4}{-HCl}$ | $M-R+H \atop -Cl$ | $RC_5H_4^+$ | $C_7H_9^+$ | $C_6H_7^+$ | $C_6H_5^+$ | Other ions |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **1** | 276 (16.2) | 241 (100) | 206 (21.4) | 204 (35.2) | 197 (46.2) | 161 (34.7) | 227 (3.6) | 79 (20.4) | 91 (−) | 79 (20.4) | 77 (44.1) | |
| **2** | 304 (3.9) | 269 (23.8) | 234 (10.5) | 232 (84.0) | 211 (62.3) | 175 (40.0) | 241 (18.9) | 93 (59.7) | 91 (99.5) | 79 (7.2) | 77 (100) | 204 (49.1) $\left(M\genfrac{}{}{0pt}{}{-2HCl}{-C_2H_4}\right)$ |
| **3** | 332 (2.9) | 297 (100) | 262 (13.5) | 260 (44.5) | 225 (32.7) | 189 (13.1) | 255 (74.3) | 107 (24.9) | 91 (27.0) | 79 (48.3) | 77 (19.2) | 218 (34.3) $\left(M\genfrac{}{}{0pt}{}{-2HCl}{-C_3H_6}\right)$ |
| **4** | 360 (2.8) | 325 (100) | 290 (16.3) | 288 (100) | 239 (43.1) | 203 (18.7) | 269 (13.9) | 121 (8.5) | 91 (16.3) | 79 (57.4) | 77 (29.7) | 232 (22.7) $\left(M\genfrac{}{}{0pt}{}{-2HCl}{-C_4H_8}\right)$ |
| **5** | 388 (2.1) | 353 (46.1) | 318 (21.0) | 316 (100) | 253 (41.5) | 217 (7.4) | 283 (12.4) | 135 (8.4) | 91 (13.4) | 79 (37.7) | 77 (22.4) | 246 (9.9) $\left(M\genfrac{}{}{0pt}{}{-2HCl}{-C_5H_{10}}\right)$ |
| **6** | 388 (0.3) | 353 (94.0) | 318 (24.8) | 316 (70.7) | 253 (19.5) | 217 (3.3) | | 135 (15.7) | 91 (25.9) | 79 (12.1) | 77 (10.8) | |
| **7** | 384 (−) | 349 (100) | 314 (22.8) | 312 (94.1) | 251 (15.0) | 215 (8.9) | 281 (10.2) | 133 (32.1) | 91 (83.7) | 79 (17.7) | 77 (14.1) | |
| **8** | 412 (−) | 377 (100) | 342 (21.8) | 340 (86.7) | 265 (12.8) | 229 (6.8) | | 147 (19.8) | 91 (24.1) | 79 (26.7) | 77 (9.5) | |
| **9** | 328 (−) | 293 (100) | 258 (10.4) | 256 (5.0) | 223 (21.0) | 187 (17.7) | | 105 (29.9) | 91 (5.4) | 79 (52.5) | 77 (61.6) | 333 (26.0) $\left(M\genfrac{}{}{0pt}{}{-Cl}{+C_3H_4}\right)$ |
| **10** | 356 (−) | 321 (100) | 286 (27.8) | 284 (6.5) | 237 (22.5) | 201 (20.0) | | 119 (16) | 91 (7.6) | 79 (13.1) | 77 (29.0) | 375 (2.6) $\left(M\genfrac{}{}{0pt}{}{-Cl}{+C_4H_6}\right)$ |
| **11** | 356 (−) | 321 (50.1) | 286 (10.6) | 284 (5.1) | 237 (100) | 201 (44.3) | | 119 (12.4) | 91 (38.3) | 79 (10.5) | 77 (23.8) | 375 (23.7) $\left(M\genfrac{}{}{0pt}{}{-Cl}{+C_4H_6}\right)$ |

| | | | | | | | [a] | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 12 | 384 (–) | 349 (100) | 314 (53.5) | 312 (9.4) | 251 (16.5) | | 216[a] (36.6) | 133 (21.5) | 91 (35.6) | 79 (9.5) | 77 (16.5) | | |
| 13 | 384 (–) | 349 (100) | 314 (69.7) | 312 (13.2) | 251 (17.7) | | 216[a] (38.6) | 133 (16.3) | 91 (17.2) | 79 (3.1) | 77 (5.2) | | |
| 14 | 412 (–) | 377 (100) | 342 (36.8) | 340 (11.4) | 265 (27.5) | | 230[a] (11.6) | 147 (5.3) | 91 (14.9) | 79 (6.8) | 77 (4.4) | | |
| 15 | 408 (–) | 373 (100) | 338 (6.7) | 336 (27.2) | 263 (100) | | 228 (84.2) | 145 (3.2) | 91 (38.9) | 79 (24.9) | 77 (27.9) | 67 (67.9) | |
| 16 | 364 (–) | 329 (100) | 294 (10.2) | 292 (1.3) | 241 (53.4) | 285 (50.2) | 205 (16.3) | | 91 (22.2) | 79 (2.0) | 77 (4.3) | 45 (37.5) (CH₃O⁺=CH₂) | |
| 17 | 392 (–) | 357 (100) | 322 (10.4) | 320 (1.2) | 255 (7.6) | | | | 91 (6.7) | 79 (0.6) | 77 (1.1) | 59 (4.8) (C₂H₅O⁺=CH₂) | |
| 18 | 564 (–) | 529 (64.9) | 494 (21.0) | 492 (3.8) | 341 (100) | | 306[a] (49.9) | 223 (20.0) | 91 (24.3) | 79 (4.1) | 77 (3.9) | 165 (19.4) | 167 (19) |
| 19 | 592 (–) | 557 (52.1) | 522 (11.2) | 520 (2.3) | 355 (100) | | 320[a] (68.4) | 237 (15.1) | 91 (2.5) | 79 (3.6) | 77 (2.7) | 105 (23.2) | 165 (16.3) |
| 20 | 592 (–) | 557 (83.4) | 522 (47.7) | 520 (7.6) | 355 (98.2) | | 320[a] (100) | 237 (29.1) | 91 (7.6) | 79 (3.9) | 77 (2.4) | 105 (30.5) | 165 (22.6) |
| 21 | 484 (–) | 449 (62.4) | 414 (14.2) | 412 (1.9) | 301 (100) | | 265 (14.9) | 183 (20.4) | 91 (13.2) | 79 (2.8) | 77 (6.8) | 153 (22.8) | 168 (19.5) |
| 22 | 512 (–) | 477 (61.4) | 422 (18.3) | 440 (2.4) | 315 (100) | | 280[a] (17.5) | 197 (10.0) | 91 (20.9) | 79 (2.5) | 77 (7.1) | 153 (24.3) | 167 (13.0) |
| 23 | 540 (–) | 505 (94.3) | 470 (35.2) | 468 (4.6) | 329 (100) | | 294[a] (26.1) | 211 (13.8) | 91 (13.2) | 79 (1.6) | 77 (4.9) | 153 (18.4) | 167 (14) |
| 24 | 544 (–) | 509 (80.9) | 474 (33.5) | 472 (4.6) | 331 (100) | | 296[a] (44.6) | 213 (61.7) | 91 (5.4) | 79 (2.2) | 77 (5.1) | 165 (18.5) | 198 (32.2) |

[a] (M – RC₅H₄ – Cl).

TABLE 2

NCI MASS SPECTRAL DATA OF COMPOUNDS **1–24** (relative intensities in parentheses)

| Compound | $M^+$ | $(M+Cl)^-$ | $(M-Cl)^-$ | $(M+Cl-RC_5H_4)^-$ | $(M+Cl-R+H)^-$ | $(M-R+H)^-$ | $(M+Cl-2R+2H)^-$ | $(M-2R+2H)^-$ | Other ions |
|---|---|---|---|---|---|---|---|---|---|
| **1** | 276 (100) | 311 (39.5) | 241 (0.6) | 232 (4.6) | 297 (2.2) | 262 (5.5) | | | |
| **2** | 304 (100) | 339 (40.4) | 269 (0.6) | 246 (7.4) | 311 (38.6) | 276 (84.0) | 283 (10.0) | 248 (7.5) | |
| **3** | 332 (90.2) | 367 (23.0) | 297 (0.5) | 260 (3.3) | 325 (27.2) | 290 (100) | 283 (11.6) | 248 (35.9) | |
| **4** | 360 (100) | 395 (26.8) | | 274 (2.3) | 339 (20.6) | 304 (97.1) | 283 (4.6) | 248 (20.6) | |
| **5** | 388 (100) | 423 (1.8) | 353 (10.5) | 288 (0.4) | 353 (10.5) | 318 (9.8) | 283 (1.6) | | |
| **6** | 388 (100) | 423 (2.3) | 353 (17.0) | 288 (0.6) | | | | | |
| **7** | 384 (100) | 419 (12.1) | 349 (8.1) | 286 (2.1) | 351 (13.7) | 316 (67.0) | 283 (1.9) | 248 (0.5) | |
| **8** | 412 (100) | 447 (4.2) | 377 (10.4) | 300 (0.6) | | | | | |
| **9** | 328 (100) | 363 (5.9) | 293 (1.3) | 258 (1.1) | | | | | 368 (17.9) $(M+R-H)$ |
| **10** | 356 (100) | 391 (3.4) | 321 (4.5) | 272 (0.8) | | | | | 410 (2.9) $(M+R-H)$ |
| **11** | 356 (100) | 391 (20.1) | 321 (2.4) | 272 (2.7) | | | | | 410 (46.0) $(M+R-H)$ |

| No. | | | | | | |
|---|---|---|---|---|---|---|
| 12 | 384 (100) | 419 (8.8) | 349 (1.6) | 286 (4.3) | | |
| 13 | 384 (100) | 419 (4.8) | 349 (2.0) | 286 (0.6) | | |
| 14 | 412 (100) | 447 (1.0) | 377 (18.9) | 300 (36.9) | 316 (3.4) | |
| 15 | 408 (100) | 443 (1.5) | 373 (69.0) | 298 (16.2) | | |
| 16 | 364 (100) | 399 (3.3) | 329 (6.0) | | | |
| 17 | 392 (100) | 427 (5.3) | 357 (7.8) | | 355 (4.2) | 320 (60.6) |
| 18 | 564 (85.9) | 599 (5.2) | 529 (100) | 376 (29.2) | | |
| 19 | 592 (29.6) | 627 (2.8) | 557 (58.3) | 390 (100) | | 500 (95) $(M - CH_3C_6H_5)$ |
| 20 | 592 (30.1) | 627 (0.1) | 557 (96.5) | 390 (100) | | 500 (20.9) $(M - CH_3C_6H_5)$ |
| 21 | 484 (49.9) | 519 (–) | 449 (100) | 336 (1.2) | | 406 (48.2) $(M - C_6H_6)$ |
| 22 | 512 (26.0) | 547 (–) | 477 (100) | 350 (–) | | 434 (1.1) $(M - C_6H_6)$ |
| 23 | 540 (84.4) | 575 (–) | 505 (100) | 364 (13.8) | | |
| 24 | 544 (62.3) | 579 (1.0) | 509 (100) | 366 (13.1) | 395 (7.4) | 436 (12.4) $(M - CH_3OC_6H_5)$ |

In an attempt to obtain a greater abundance of ions characteristic of the molecular weight, we determined the NCI mass spectra of compounds **1–24** using $CH_4$ as the reagent gas. A selection of our results is presented in Table 2. The major ions observed in all spectra are molecular ions, occurring mostly as base peaks. Thus, the $CH_4$ negative ion chemical ionization (NCI) mass spectra are preferred for providing molecular weight information. In addition to forming molecular ions, the NCI mass spectra of compounds **1–24** exhibited a series of ions giving structural information: $(M + Cl)^-$, $(M - Cl)^-$ and $(M + Cl - RC_5H_4)^-$ (except **16**, **17**). For compounds **1–5**, **7** and **17**, the ions $(M + H - R)^-$, $(M + 2H - R)^-$, $(M + Cl - R + H)^-$ and $(M + Cl - 2R + 2H)^-$ were observed. The adduct ion $(M + Cl)^-$ was formed by an ion-molecule reaction, because the chloride ion may have acted as a nucleophile in the gas phase. The adduct ion $(M + C_nH_{2n-2})^-$ $(n = 3-4)$ were observed in the NCI mass spectra of compounds **9–11**. Similar adduct ions $(M - Cl + C_nH_{2n-2})^+$ were seen in the EI mass spectra. The other ions $(M - CH_3C_6H_5)^-$, $(M - C_6H_5)^-$, $(M - C_6H_5 - C_3H_5)^-$ and $(M - CH_3OC_6H_5)$ were also observed for compounds **19–24**, respectively.

The NCI technique is a successful and simple method for determining the molecular weight and for obtaining valuable structural information of organometallic compounds.
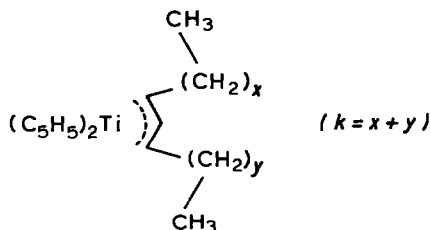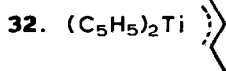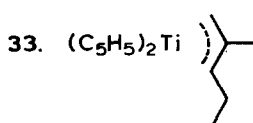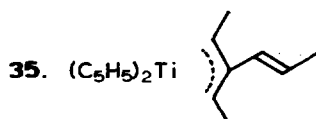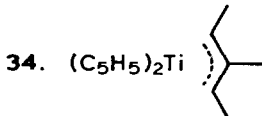
At present little is known about allyl complexes of titanium(III) or of the other oxidation states of titanium. The compounds $(C_5H_5)_2TiR$ (R = allyl, 1-methylallyl, 2-methylallyl, 1,3-dimethylallyl and 1,1-dimethylallyl), are extremely air-sensitive complexes [5]. The infrared spectra indicate [5] that the allyl ligands are $\pi$-bonded

TABLE 3

EI MASS SPECTRAL DATA OF COMPOUNDS **25–35** (relative intensities in parentheses)

| Compound | $M^+$ | $(C_5H_5)_2Ti^+$ | $C_5H_5Ti^+$ | Other ions |
|---|---|---|---|---|
| **25** | 261 | 178 | 113 | 152, 87, 73, 48 |
| | (21.0) | (100) | (53.8) | |
| **26** | 275 | 178 | 113 | 96, 81, 67, 55, 41 |
| | (7.0) | (100) | (33.0) | |
| **27** | 289 | 178 | 113 | 152, 87, 81, 55, 41 |
| | (9.0) | (100) | (13.0) | |
| **28** | 317 | 178 | 113 | 140, 95, 81, 69, 55, 41 |
| | (7.0) | (100) | (17.0) | |
| **29** | 331 | 178 | 113 | 152, 81, 67, 41 |
| | (4.0) | (100) | (28.0) | |
| **30** | 345 | 178 | 113 | 166, 111, 97, 83, 69, 55, 41 |
| | (31) | (89) | (–) | |
| **31** | 373 | 178 | 113 | 194, 95, 81, 67, 55, 41 |
| | (4.0) | (100) | (19.0) | |
| **32** | 233 | 178 | 113 | 87, 73, 55, 41 |
| | (9.5) | (100) | (21.6) | |
| **33** | 261 | 178 | 113 | 152, 87, 71, 57, 41 |
| | (6.4) | (100) | (20.0) | |
| **34** | 261 | 178 | 113 | 87, 67, 55, 41 |
| | (7.7) | (100) | (31.0) | |
| **35** | 287 | 178 | 113 | 108, 93, 77, 53, 41 |
| | (7.1) | (100) | (12.9) | |

to the metal, as are the cyclopentadienyl ligands. The mass spectra of allyl complexes of Ti, Nb and Ta together with the methylallyl complexes of Ta were reported in 1974 [6]. The fragmentation patterns of the complexes $(C_5H_5)_2MR$ ($M$ = Ti, Nb, Ta) do not depend strongly on the nature of the metal or on the position of the methyl group. In all cases fragmentation starts with elimination of the allyl group. Recently alkenyldicyclopentadienyltitanium complexes have received attention [7]. We have studied the EI mass spectra of allyldicyclopentadienyltitanium(III) for compounds **25–35** and in all cases the molecular ion peaks



| | | |
|---|---|---|
| **25.** $k = 1$ | **29.** $k = 6$ | **32.** $(C_5H_5)_2Ti$ |
| **26.** $k = 2$ | **30.** $k = 7$ | **33.** $(C_5H_5)_2Ti$ |
| **27.** $k = 3$ | **31.** $k = 9$ | |
| **28.** $k = 5$ | | **34.** $(C_5H_5)_2Ti$ |
| | | **35.** $(C_5H_5)_2Ti$ |

were observed, with low intensities. The prominent ions are listed in Table 3. The fragmentation pattern of 1,3-disubstituted allyldicyclopentadienyltitanium complexes is similar to that of the $Cp_2Ti(\pi$-allyl) complex. The mass spectra of compounds **25–35** show that the molecular ions decompose via two routes resulting in the elimination of R and $(C_5H_5)_2Ti$ and the formation of $(C_5H_5)_2Ti^+$ and $R^+$. The major metallic ions are formed by loss of $C_5H_5$ or $C_2H_2$ from $(C_5H_5)_2Ti$, and $(TiC_3H_3)^+$ was formed from $(C_5H_5TiC_3H_3)^+$ by elimination of $C_5H_5$. The $R^+$ ions lose H or 2H to give $(C_nH_{2n})^+$ and $(C_2H_{2n-3})^+$.

## Experimental

The mass spectra were recorded on Finnigan 4021 quadrupole mass spectrometers. Methane was used as the NCI reagent gas at a pressure of 0.3 Torr. The source

temperature was 200°C for NCI and 250°C for EI. The solid insertion probe was used at 50–250°C. Some of the data of allyldicyclopentadienyltitanium compounds were recorded on Varian Mat 311 at the Max-Planck Institut für Kohlenforschung in West Germany.

## Acknowledgement

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