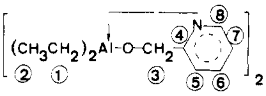
Table I. ^1H, ^{13}C, and ^{27}Al NMR Signal Assignments of 3 at Various Temperatures

	position for numbering								
	1A	1B	2	3	4	5	6	7	8
					$^1H^a$				
δ	0.57	0.46	1.52	4.85		5.97	6.64	6.33	8.17
multiplicity	dq	dq	q	s		d	dd	dd	d
J, Hz	13.6 8.0	13.6 8.0	8.0			7.9	7.9 7.4	7.4 5.4	5.4
					$^{13}C^b$				
δ	2.21		11.16	63.00	159.11	119.76	137.60	122.38	144.91
multiplicity	t		q	t	s	d	d	d	d
$^1J(C,H)$, Hz	115		124.0	142.5		166.0	164.0	167.0	181.5
					^{27}Al				

	$3^{c,f}$				background signal^d	
T	310	300	273	243	273	243
δ	106	108	109	~110	58	62
$W_{1/2}$ Hz	5400	6200	10000	e	5800	6000

[a] C_7D_8; $T = 213$ K; 5 mm sample, ~1%. [b] C_7D_8; $T = 273$ K; 10 mm sample, ~8%. [c] C_7D_8; T, cf. Table I; 10 mm sample, ~8%. [d] Present also in spectra of samples containing no ^{27}Al nuclei. [e] $W_{1/2}$ not determined exactly. [f] Atom numbering:



3

pendent of the temperature between 310 and 243 K. Due to the reduced solubility of **3** and to the increasing line width ($W_{1/2}$ of **3** at 273 K is already about 10000 Hz) with decreasing temperature, its ^{27}Al NMR signal below 243 K is difficult to detect. The temperature dependence of $W_{1/2}$ of **3** is normal (cf. Table I) ($W_{1/2}$ increases with decreasing temperature. Note that according to ref 1 $W_{1/2}$ at 223 K should be smaller than that at 295 K!). In line with a fivefold coordinated Al atom in **3** the α-protons of the terminal ethyl groups are diastereotopic between 213 and 273 K. Revised assignments of the observed ^{27}Al and ^1H NMR spectra of **3** are also presented in Table I together with the ^{13}C NMR signal assignments as obtained from a two-dimensional ^{13}C, ^1H shift correlated spectrum.[4]

From our findings we conclude that there is no significant temperature dependence of the ^{27}Al NMR shifts of **1** between 310 and 223 K nor of **2** and **3** between 310 and 253 K and between 310 and 243 K, respectively, and these spectra provide no evidence for the existence of an equilibrium between four- and five-coordinated aluminum atoms. The ^1H NMR spectra of **3**, however, indicate that dissociation occurs to some minor extend at temperatures above 310 K (cf. also ref 1). Moreover, caution is necessary when NMR spectra of dilute samples of broad quadrupolar metal nuclei are recorded, since there are many causes for background signals.[5] Nevertheless, in spite of these difficulties, NMR spectroscopy of metal nuclei[5,6] is extremely useful for investigating many problems in organometallic chemistry.

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(4) The assignments of $^2J(H_A,H_B)$ and $\delta(H_6)$ and $\delta(H_7)$ in Table I differ from those given in ref 1. A comprehensive paper on the preparation and the NMR spectra of organoaluminium compounds is in preparation: Benn, R.; Janssen, E.; Lehmkuhl, H.; Rufińska, A., to be submitted for publication.

(5) Benn, R.; Rufińska, A. *Angew. Chem.* **1986**, *98*, 000.

(6) (a) Kidd, R. G. *Annu. Rep. NMR Spectrosc.* **1980**, *10A*, 1. (b) Dechter, J. J. *Prog. Inorg. Chem.* **1982**, *29*, 285. (c) Rehder, D. *Magn. Reson. Rev.* **1984**, *9*, 125.

Additions and Corrections

John H. Freudenberger and Richard R. Schrock*: Preparation of Di-*tert*-butoxytungsten(VI) Alkylidene Complexes by Protonation of Tri-*tert*-butoxytungsten(VI) Alkylidyne Complexes. **1985**, *4*, 1937.

The correct title is published above. The title was incorrectly published as "Preparation of Di-*tert*-butoxytungsten(VI) Alkylidene Complexes by Protonation of Tri-*tert*-butoxytungsten(VI) Alkylidene Complexes".