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The Chemical Reactivity of Tetraisobutyldialuminum Towards Ethers, Nitrogen Lewis Bases, Aluminumtrimethyl, Borontrichloride and Ethylene

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Abstract

Treatment of $Al_2{}^iBu_4$ with THF and Et_2O results in partial decomposition to afford Al metal while reaction with γ-picoline produces a bis-adduct of limited stability. Reactions with $AlMe_3$ and BCl_3, separately, involves ligand exchange with accompanying disproportionation to yield Al metal. Dimethylamine induces disproportionation to afford $Al^iBu_3 \cdot HNMe_2$ and an intermediate trialuminum species. The latter undergoes Al–Al bond cleavage with formation of H_2, ${}^iBu_2AlNMe_2$, and $[Me_2N({}^iBu)Al{-}Al^iBu_2]_2$. $Al_2{}^iBu_4$ eliminates $Me_2C{=}CH_2$ in solution at 80 °C, and the catenated Al–H intermediate reacts with ethylene to afford AlEt and $AlCH_2CH_2CH_2CH_3$ moieties.

Introduction

We have previously reported the chemical behavior of tetraisobutyldialuminum, $Al_2{}^iBu_4$, **1**, towards a variety of protonic reagents and observed that the Al–Al bond is cleaved more easily than the Al–C bond [1]. In addition, **1** undergoes a slow decomposition in nonpolar solvents as evidenced by the deposition of Al metal. The rate of this reaction is qualitatively dependent on the solvent employed, the solution temperature, and possibly the concentration of **1**. On the other hand, neat **1** is stable at 40 °C, while solution of **1** evolves H_2 at 80 °C. It would appear the steric bulk of the ${}^iBu$ moiety inhibits disproportionation, perhaps involving an intramolecular 1 to 2 ${}^iBu$ shift. It should be noted that alkyl exchange between **1** and $AlEt_3$ affords Al metal [2], that is, non-sterically hindered alkyls do not result in stable catenated Al derivatives.

In an effort to further develop an understanding of the factors involved in the reactivity of the Al–Al bond, **1** was treated with THF, Et_2O, γ-picoline, $HNMe_2$, BCl_3, $AlMe_3$, and C_2H_4.

Results and Discussion

When a cyclopentane solution of **1** is maintained at 25 °C for several days, Al metal is recovered by filtration and subsequently identified as the 8-hydroxyquinolate. In an effort to inhibit decomposition of **1**, by coordination, cyclopentane solutions were separately treated with excess THF and Et_2O. In both cases an increased rate of decomposition of **1** was noted with formation of Al metal in 1 h. The bis-THF adduct of **1** has been reported as a result of reaction between $K_2Al_2({}^iBu)_6$ and Me_3SiCl in THF, however, no characterization data were presented [3].

We have observed the formation of **1**·(γ-picoline)$_2$, **2**, upon treatment of a benzene solution of **1** with excess γ-picoline with the stoichiometry based on integration of the ${}^1H$ NMR absorptions, $\mathit{CH}_3C_5H_4N$: CH_2CHMe_2; found, 1.28; calc. 1.33. Calc. for the monoadduct, 2.66. However, **2** is also unstable in benzene as evidenced by formation of a precipitate after one day at 25 °C.

With Lewis Acids

Treatement of **1** with excess BCl_3 results in ${}^iBuCl$ exchange and accompanying decomposition of the dialuminum exchange product. The red–brown color of **1** is discharged after two weeks and Al metal may be recovered after several days. The ${}^1H$ NMR spectrum of the initial reaction solution exhibits a doublet centered at 0.93 ppm assigned to the B–${}^iBu$ moiety ($CH_2CH\mathit{Me}_2$) with reference to B^iBu_3, 0.98 ppm [4]. A less intense, *ca.* 1/6, doublet centered at 1.24 ppm is assigned to an Al–${}^iBu$ species (Me_2CHCH_2) which compared with $({}^iBu)_2AlNMe_2$ (1.21d), (Bu_3Al (1.11d), and $Al_2{}^iBu_4$ (1.16d). Hydrolysis of an aliquot of this solution, previously filtered to remove Al metal, affords isobutane and H_2 in 13.8:1 mole ratio respectively indicating extensive Al–Al bond cleavage has occurred; found for **1**, 4:1 respectively. The B–${}^iBu$ moiety does not afford isobutane under the reaction conditions employed.

The result of ligand exchange between **1** and $AlMe_3$ is similar to that reported for BCl_3, *i.e.*, the

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Al–Al bond is destroyed. **1** was treated with $AlMe_3$, 1:2.46 mole ratio respectively, and Al metal precipitated from solution after several hours. Hydrolysis of a filtered aliquot of this solution would be expected to afford H_2:${}^iBuH$:CH_4, in the respective ratios, 1:4.0:4.2, based on the relative amounts of reagents employed (H_2:total alkyl, 1:8.2). The observed molar ratios are 1:19.4:20.2 respectively, (H_2:total alkyl, 1:40), with the CH_4:${}^iBuH$ observed 1.04, calc. 1.04. The aliquot subjected to hydrolysis contained 35% of the initially present ${}^iBu$ moieties, but only 3% of the initial Al–Al bonds. Because the above observed CH_4:${}^iBuH$ ratio is that calculated, it is apparent that the decomposition of **1** involved a disproportionation, eqn. (1), and not a reaction involving the ${}^iBu$ moiety.

$$3Al_2{}^iBu_4 \longrightarrow 4Al{}^iBu_3 + 2Al \tag{1}$$

It is interesting to note that the analogous reverse of this reaction, employing K metal, has been reported to afford the catenated Al species $K_2Al{}^iBu_6$ [5]. Furthermore, when **1** is prepared from ${}^iBu_2AlCl$ and K, we routinely observe a 10% yield of $Al{}^iBu_3$. That is, one must take care, during the purification of **1**, to remove $Al{}^iBu_3$ as well as establish the absence of unreacted ${}^iBu_2AlCl$.

With Dimethylamine

In an effort to prepare $Al_2(NMe_2)_4$, analogous to $B_2(NMe_2)_4$, **1** was treated with $HNMe_2$ at 0, 25, and 43 °C. Rather than ligand exchange, to be expected at elevated temperatures [6], H_2 is evolved even at 0 °C. One may explain H_2 evolution in terms of nucleophilic attack by $NHMe_2$ at Al and generations of the Al–H moiety, eqn. (2), followed by elimination of H_2, eqn. (3). However, based on this reaction

$${}^iBu_2Al{-}Al{}^iBu_2 + HNMe_2 \longrightarrow {}^iBu_2AlH + {}^iBu_2AlNMe_2 \tag{2}$$

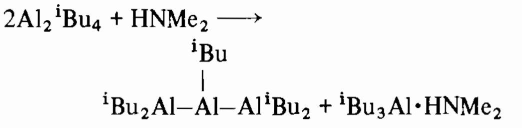
$${}^iBu_2AlH + HNMe_2 \longrightarrow H_2 + {}^iBu_2AlNMe_2 \tag{3}$$

sequence only a 50% yield of H_2 is obtained after 100 h at 25 °C (*ca.* 40% after 2 h), 50% after 20 h at 43 °C and 60% after 40 h at 43 °C. A solution of **1**, maintained at 25 °C in the presence of excess $NHMe_2$ ($HNMe_2$ was recovered after reaction), afforded a 55% H_2 yield, eqn. (4). The indicated residue (its red–brown color is indicative of catenated Al), was filtered to remove Al metal resulting from disproportionation, and was subjected to hydrolysis, eqn. (5). A 100% yield of Al–${}^iBu$ moieties was obtained while additional H_2 evolution, eqn. (5), indicated the presence of a catenated Al species relatively unreactive toward $HNMe_2$ compared to **1**. The total H_2 yield, eqns. (4) and (5), (*ca.* 90%) is consistent with partial decomposition of **1**, eqn. (1).

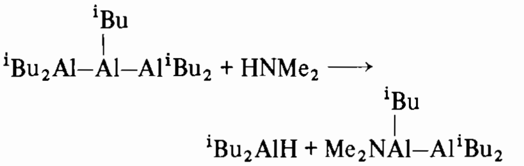
$$Al_2{}^iBu_4 + HNMe_{2(exc)} \longrightarrow 0.55H_2 + \text{residue} \tag{4}$$

$$\text{residuc} + H_3O^+_{(exc)} \longrightarrow 0.33H_2 + 4.07{}^iBuH \tag{5}$$

It is suggested that $HNMe_2$ not only functions to cleave Al–Al bonds, but in a prior step, induces disproportionation, eqn. (6), followed by a cleavage reaction, represented by eqn. (7), and H_2 evolution, eqn. (8) (50% yield of H_2), based on the total number of Al–Al bonds.



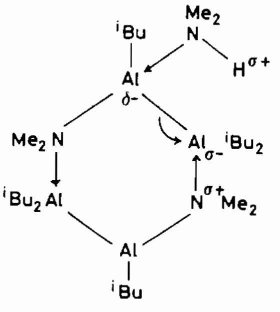
$$2Al_2{}^iBu_4 + HNMe_2 \longrightarrow {}^iBu_2Al{-}\overset{{}^iBu}{\overset{|}{Al}}{-}Al{}^iBu_2 + {}^iBu_3Al{\cdot}HNMe_2 \tag{6}$$



$${}^iBu_2Al{-}\overset{{}^iBu}{\overset{|}{Al}}{-}Al{}^iBu_2 + HNMe_2 \longrightarrow {}^iBu_2AlH + Me_2N\overset{{}^iBu}{\overset{|}{Al}}{-}Al{}^iBu_2 \tag{7}$$

$${}^iBu_2AlH + HNMe_2 \longrightarrow {}^iBu_2AlNMe_2 + H_2 \tag{8}$$

One would expect the suggested catenated Al product, **3**, eqn. (7), to exist on an N-bridged cyclic dimer and therefore to sterically resist nucleophilic attack by additional $HNMe_2$. Furthermore, oxidation of an Al–Al bond, **3**, would be expected to be slow because heteronuclear Al–Al bond cleavage, affording Al^{1-} (necessary for subsequent protonation by $HNMe_2$) would not be favored due to excess negative charge, on that same Al atom, as a result of coordinated covalent bonding by the NMe_2 moiety, *i.e.*:



3

The ${}^1H$ NMR spectrum of the initial reaction mixture, 30% to 50% H_2 evolution, consists of absorptions at 2.21(s), 1.76(s), 1.69(s), 1.24(d), 0.20(d), and 0.04(d) ppm in the approximate ratios 2:2:2.1:20:0.8:4, respectively. The postulated disproportionation product, $Al{}^iBu_3{\cdot}HNMe_2$, eqn. (6), was independently prepared and exhibited ${}^1H$ NMR absorptions at 1.76(s), $Me_2NH \rightarrow Al$, 1.21(d) Me_2CHCH_2, and 0.06(d) ppm, Me_2CHCH_2 with thc expected *Me* ratios of 1:3 respectively. Therefore the absorptions in the reaction mixture, at 1.76(s) and 0.04(d), are indicative of the presence of Al(^{i}Bu)$_3$·HNMe$_2$. Furthermore, (^{i}Bu)$_2$AlNMe$_2$ was independently prepared and exhibited absorptions at 2.24(s), *Me*$_2$N-Al, 1.21(d), *Me*$_2$CHCH$_2$, Me$_2$CH*CH*$_2$, and 0.25(d) ppm, in the expected ratios. The absorption at 2.21(s) ppm, in the reaction mixture, is therefore consistent with the presence of the Me$_2$N–Al moiety, eqn. (8). Finally, after 50% to 55% H$_2$ evolution, the reaction solution was heated at 60 °C for 2 days, 10^{-5} torr, to remove Al^{i}Bu$_3$·HNMe$_2$ and ^{i}Bu$_2$AlNMe$_2$. The red–brown reaction residue was filtered, employing cyclopentane, and the filtrate exhibited ^{1}H NMR absorptions at 2.18(s), *Me*$_2$N–Al and 1.15(d), *Me*$_2$CHCH$_2$–Al in the ratios 1:3 respectively, consistent with the suggested formulation for **3**. Concerning the ^{1}H NMR spectrum of the initial reaction mixture, the absorption at 1.69(s) ppm is indicative of coordinated HNMe$_2$, perhaps weakly associated with **3** in solution.

To summarize this complicated chemical system 50% of the Al–Al bonds are rapidly oxidized by HNMe$_2$ and the remaining solution contains catenated aluminum, perhaps in the form suggested by **3**, ^{i}Bu$_3$Al·HNMe$_2$, and ^{i}Bu$_2$AlNMe$_2$.

With Ethylene

A solution of **1** was slowly heated to 80 °C with periodic examination for the presence of volatile decomposition products. H$_2$ evolution commences at 80 °C while maintaining **1** at 125 °C for 10 h affords isobutylene and H$_2$, in a 2:1 molar ratio respectively, with discharge of the red–brown solution color. This chemical transformation involves the well-known β-H elimination and thermal decomposition of the generated Al–H moiety, eqn. (9).

$$\mathrm{Al{-}^{i}Bu \longrightarrow [Al{-}H] + Me_2C{=}CH_2} \qquad (9)$$

$$\mathrm{\hookrightarrow Al + \frac{1}{2}H_2}$$

It is not clear whether this reaction, at 125 °C, involves Al^{i}Bu$_3$, thermally generated, eqn. (1), or **1**; in both cases the expected Me$_2$C=CH$_2$:H$_2$ ratio is 2. Catenated Al is present at 80 °C based on the solution color, dark red–brown.

In an effort to establish the formation of a catenated Al hydride as a thermolysis intermediate, a solution of **1**, maintained at 80 °C was periodically treated with increments of ethylene, total pressure 200 to 600 torr, over a period of 32 days. The overall stoichiometry associated with this reaction is summarized by eqn. (10).

$$\begin{aligned}\mathrm{Al_2{}^{i}Bu_4 + 6C_2H_4} &\longrightarrow \mathrm{3Me_2C{=}CH_2}\\ &\quad + \text{red–brown residue}\\ &\quad + \text{low-volatile clear liquid} \qquad (10)\end{aligned}$$

The red–brown reaction residue, filtered employing benzene, does not contain the Al–H moiety based on the lack of an infrared absorption at 1800 cm^{-1} [7]. However, hydrolysis of this material affords H$_2$, C$_2$H$_6$, and n-butane in equal molar amounts. Based on the color of this material, and the fact that H$_2$ is evolved during hydrolysis, there is little doubt that this material still contains catenated Al. Furthermore, the evolution of both C$_2$H$_6$ and n-butane during hydrolysis establishes both C$_2$H$_4$ insertion into a molecular catenated Al–H species and C$_2$H$_4$ insertion into the Al–Et bond. Based on the relative rates of C$_2$H$_4$ uptake and isobutylene evolution, insertion into the Al–Et bond is a more facile reaction than isobutylene elimination.

The colorless liquid product, eqn. (10), was not fully characterized but is most likely a mixed trialkyl aluminum species; the ^{i}Bu moiety is absent based on the lack of a septet at *ca.* 2.1 ppm (Me$_2$*CH*CH$_2$), and does not contain the Al–H function based on the absence of the corresponding absorption for ~1800 cm^{-1}.

Experimental and Techniques

Standard vacuum line procedures were employed throughout this investigation [1]. Infrared spectra were recorded with a Perkin-Elmer 457 spectrometer with non-volatile samples prepared neat or as Nujol and Fluorolube mulls with KBr plates. Volatile samples were contained in a 10 cm gas cell. ^{1}H NMR spectra were recorded with a JEOL E360 spectrometer employing the solvent as an internal reference, while chemical shifts were measured with respect to TMS. Quantities of H$_2$ were determined, after collection in a calibrated Toepler pump system, by combustion to H$_2$O over CuO at 300 °C.

Reagents

Tetraisobutyldialuminum, Al$_2{}^i$Bu$_4$, was prepared by the literature method [2]. Solvents were dried over LiAlH$_4$ and distilled prior to use.

^{i}Bu$_2$AlCl

Texas Alkyls, Inc., was used as received.

(^{i}Bu)$_3$Al

Recovered from the synthesis of Al$_2$(^{i}Bu)$_4$ and characterized by its vapor pressure, ^{1}H NMR and IR spectrum.

(^{i}Bu)$_3$Al·HNMe$_2$

Prepared by treating a benzene solution of (^{i}Bu)$_3$Al with excess HNMe$_2$. After removal of the solvent and excess HNMe$_2$, the colorless non-volatile liquid (25 °C/10^{-5} torr) exhibited ^{1}H NMR absorptions at 1.76(s) (Me_2N) and 1.21(d) ppm ($(CH_3)_2CHCH_2$), in the expected ratio, 1:3 respectively, and at 0.06(d) ppm.

$(^iBu)_2AlNMe_2$

Prepared by treating $(^iBu)_2AlH$, in benzene, with an excess of $HNMe_2$ at 25 °C. H_2 was evolved and after removal of all materials volatile at 25 °C/10^{-5} torr, a benzene solution exhibited ^1H NMR absorptions at 2.24(s) (Me_2N) and 1.21(d) (Me_2CHCH_2) ppm in expected ratio 1:2 respectively.

Trimethylaluminum

Ethyl Corp., was used as received.

Borontrichloride

The Matheson Co., was purified by fractional condensation through a −78 °C trap and collected at −126 °C.

4-Picoline

Reagent grade, Eastman Kodak Company, was dried over CaH_2 and distilled prior to use.

Dimethylamine

The Matheson Co., was dried over P_2O_5 at 0 °C and purified by fractional condensation.

Ethylene

The Matheson Co., was dried by passage to a trap maintained at −126 °C.

Reactions of $Al_2(^iBu)_4$ **1**

With BCl_3

A benzene solution of **1**, 0.282 g (1.00 mmol) was treated with 4.88 mmol BCl_3 in *ca.* 1 mmol increments. After each addition, at −196 °C, the mixture was warmed to 25 °C and stirred for 24 h. Prior to addition of the second and subsequent aliquots, the vapor pressure was checked for excess BCl_3. A grey precipitate, subsequently identified as Al(0), was observed after the first increment of BCl_3 and increased in amount as the reaction proceeded. After reaction the volatile material was removed and the residue pumped on for 30 min/10^{-5} torr. Benzene and BCl_3, 0.81 mmol, were recovered. The reaction residue, a brown oil, and Al(0) were separated by filtration employing benzene. An aliquot of the filtrate was treated with H_2O to afford H_2, 0.19 mmol, and isobutane, 2.75 mmol.

With $AlMe_3$

1, 0.168 g (0.594 mmol), dissolved in 10 ml cyclopentane was cooled to −196 °C and treated with $AlMe_3$, 0.821 mmol. The mixture was slowly warmed to room temperature; after 2 h Al(0) was observed. The mixture was stirred at 0 °C for 10 h during which time additional Al(0) precipitated, however the solution remained dark red–brown. The solution was filtered and the recovered Al(0) dissolved with aqueous HCl and subsequently isolated as the 8-hydroxyquinolate. An aliquot of the filtrate was treated with H_3O^+ to afford H_2, 0.05 mmol, CH_4, 1.01 mmol, and isobutane, 0.97 mmol, with the latter separated from H_2O by passage through a −95 °C trap and characterized by its IR spectrum. The remainder of the reaction solution became colorless after 1 week, indicating the absence of catenated Al.

With Lewis Bases

1, *ca.* 0.1 g was treated with 5 ml Et_2O at room temperature. After 1 h Al(0) precipitated from solution and was removed by filtration. The filtered solution was stirred for 10 additional hours and again Al(0) resulted. Similar results were obtained with THF. **1**, 0.156 g (0.55 mmol), was dissolved in 5 ml benzene and treated with *ca.* 1 ml 4-picoline. After stirring the mixture for 15 min at 25 °C, the volatile materials were removed and the residue pumped on, 10^{-5} torr, for 9 h. The non-volatile, dissolved in benzene, exhibited ^1H NMR absorptions centered at 1.69(s), ($\underline{Me}C_5H_4N$), 1.4(d) ($\underline{Me_2}CHCH_2$), and 0.56(d) ($Me_2CH\underline{CH_2}$) ppm. After one day decomposition was evidenced by precipitate formation.

With $HNMe_2$

1, 0.3184 g (1.13 mmol) in 15 ml cyclopentane was treated with 2.20 mmol $HNMe_2$ at −196 °C and the reaction mixture subsequently stirred for 2h at 0 °C. H_2, 0.35 mmol was liberated and fractionation of the reaction mixture afforded cyclopentane and $HNMe_2$ both of which were separately characterized by their respective IR spectra. These materials were returned to the reaction mixture and the contents stirred at 25 °C for an additional 20 h to produce 0.13 mmol H_2, total 0.48 mmol. During both reaction periods no isobutane was evolved. An additional 2.15 mmol $HNMe_2$ was added to the reaction mixture which was stirred at 25 °C for 6.8 days to produce 0.075 mmol H_2 (total, 0.55 mmol). After an additional 27 days of reaction at 25 °C, 0.15 mmol H_2 was evolved. After this reaction period the dark red–brown reaction mixture was treated with H_3O^+ to afford H_2, 0.31 mmol and isobutane, 4.60 mmol. In a separate reaction **1**, 0.94 mmol was treated with 3.75 mmol $HNMe_2$ and the reaction mixture maintained at 43 °C. The total amount of H_2, mmol, evolved as related to total reaction time (days) were respectively as follows: 0.19, (0.08); 0.49, (0.75); 0.52, (1.3); 0.56, (2.2); 0.59, (3.8); 0.61, (5.5); and 0.63, (7.2). These data indicate a relatively rapid H_2 evolution, 6.5×10^{-1} mmol day^{-1}, followed by slow continuous H_2 evolution, 1.2×10^{-2} mmol day^{-1} or the initial rate is

54 times that of the latter rate. No isobutane was evolved during this reaction at 43 °C. In a separate reaction, **1**, 0.24 mmol was treated with $HNMe_2$, 1.02 mmol, in toluene for 3 days at 40 °C followed by removal of all volatile materials at 40 °C/10^{-5} torr for 3 days. The red–brown reaction residue was filtered with cyclopentane and exhibited ^1H NMR absorptions at 2.18 ($\underline{Me_2}N$), 1.15(d), ($\underline{Me_2}CHCH_2Al$), and 0.20 ppm, ($Me_2CH\underline{CH_2}Al$)) with the first two absorptions in the ratio 1:3 respectively.

Thermolysis of 1

A sample of **1**, in toluene, was heated to 83 °C over a period of 1.5 days with periodic cooling to −196 °C. H_2 evolution was noted, 80–83 °C. Next the toluene was replaced with *o*-xylene and the sample maintained at 125 °C for 3 additional days after which time the solution was colorless. H_2, 0.21 mmol and isobutylene 0.41 mmol were evolved; the latter was identified by comparison of its IR spectrum with an authentic sample.

With ethylene

In a typical reaction **1**, 1.34 mmol, dissolved in toluene, was treated with increments of C_2H_4, typically 1.5 mmol (total pressure ~600 torr), and the reaction mixutre maintained at 80 °C for varying time periods, 12 h initially to 7 days finally. A total of 12 C_2H_4 additions were carried out, and after each reaction period the volatile materials were separated employing traps maintained at −196, −126 and −78 °C. Ethylene was recovered from the −196 °C trap while isobutylene was collected in the −126 °C trap.

During the initial 12 h reaction period, C_2H_4 was absorbed at a rate ~6 times greater than that of isobutylene evolution (1.3×10^{-2} mmol h^{-1}). After 91 h of reaction the rates were comparable, ~2.5×10^{-2} mmol/h. The final reaction period of 3 days, (total reaction time, 32 days), involved C_2H_4 absorption at 5.4×10^{-3} mol h^{-1} and isobutylene elimination at 1×10^{-3} mmol h^{-1}. After 32 days a total of 8.13 mmol C_2H_4 were absorbed and 3.98 mmol isobutylene evolved. The reaction residue consisted of a non-volatile red viscous material; a non-volatile colorless liquid was observed in an adjacent trap which was carried from the reaction vessel during solvent vaporization. An infrared spectrum of both materials indicated the absence of an Al–H moiety, *i.e.*, ν(Al–H) at ~1800 cm^{-1}. Acid hydrolysis of an aliquot of the colored residue, previously filtered employing benzene, afforded H_2, 0.67 mmol; C_2H_6, 0.63 mmol, and n-butane, 0.63 mmol. The latter was characterized by comparison of its IR spectrum with an authentic sample. The ^1H NMR spectrum of the colored residue, benzene solution, was poorly resolved even on dilution, and exhibited broad absorptions centered at 1.45, 1.12, and 0.18 ppm. The spectrum of the colorless liquid contained broad multiple absorptions in the range 1.76–0.76 ppm and 0.66–0.06 ppm.

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