# The reactions of nitromethane in the gas phase and on a Co-ZSM5 catalyst under the conditions of the methane / $NO_x$ SCR reaction

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The rate of reaction of nitromethane in the gas phase is little changed by the presence of $O_2$ or NO. The major products are CO, NO and $H_2O$, plus $NO_2$ if $O_2$ is included. The corresponding catalytic reaction over fresh Co-ZSM5 commences at a much lower temperature and gives primarily $CO_2$ and $NH_3$, the latter being oxidised to $N_2$ above 340°C. As the catalyst ages below 320°C HNCO eventually becomes the major nitrogen-containing product although it can be hydrolysed by added water. The results demonstrate one possible route for the methane-SCR reaction.

**Keywords:** nitromethane, Co-ZSM5, $NO_x$ reduction by methane, selective catalytic reduction

## 1. Introduction

There is much current interest in the selective catalytic reduction (SCR) of nitrogen oxides by hydrocarbons in the presence of oxygen as a way of reducing $NO_x$ emissions from lean-burn engines and power stations. The most studied systems are Cu-ZSM5, using $C_{2+}$ hydrocarbons [1] and Co-ZSM5 with methane [2] but there is still uncertainty about the mechanism in both cases. In the latter case the onset of reaction seems associated with $NO_2$ formation [3]. The reaction exhibits a large deuterium kinetic isotope effect [4] consistent with the view that the rate-limiting step is hydrogen abstraction, possibly by adsorbed $NO_2$ [5–7], to form a methyl species. This has led to suggestions that subsequent reactions of $CH_3$ with $O_2$ and $NO_x$ play a role in SCR chemistry [3,7,8].

It is known that small amounts of NO greatly reduce the temperature at which methane reacts with oxygen in the gas phase [9]. However, the product of the process is $NO_2$ not $N_2$ as achieved by SCR. In recent work Bromly et al. [9] have described a kinetic model which can reproduce the features of the homogeneous reaction. Nitromethane, formed by addition of $CH_3$ and $NO_2$, is a significant species in the model. In this context several authors have suggested that nitromethane could also be formed by coupling of $CH_3$ and $NO_2$ during the methane-SCR reaction over Co-ferrierite and Co-ZSM5 catalysts and proposed steps by which it might be converted to nitrogen [5,7,8]. There is no literature report of this proposal being examined experimentally but several tests made in connection with proposals that the SCR reaction using higher hydrocarbons over other zeolites proceeds via nitrocompounds indicate that it is possible with Ce-ZSM5 [10], Cu-ZSM5 [10,11] and H-ZSM5 [11]. By contrast, the decomposition of nitromethane over many metal-exchanged X and Y zeolites yields largely ammonia and carbon dioxide [12].

In this study we have determined the products of the oxidation of nitromethane over a Co-ZSM5 catalyst and in the gas phase. Prior results for the latter are confined to conditions very different to those employed for SCR [13–16]. Our results establish one sequence of steps through which nitromethane could participate in the production of nitrogen in the methane-SCR reaction although it is unclear if it is the sole one.

## 2. Experimental

### 2.1. Gas phase experiments

Nitromethane was oxidised by passing reactant mixtures, at flow rates of 100 ml/min, through a laminar flow reactor consisting of a quartz tube of 5 mm inside diameter placed in a tube furnace. Reactant flows were prepared at nominal concentrations of 320 ppm nitromethane, 3% oxygen and 150 ppm NO (where applicable) in helium to a total pressure of 1 atm. These

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concentrations were prepared by diluting more concentrated mixtures of the gases using Tylan electronic mass flow controllers, calibrated using a Buck MP5 soap film flow meter. Nitromethane flows were diluted from approximately 2500(±300) ppm nitromethane in He mixtures which were prepared manometrically from degassed laboratory grade nitromethane.

Product and reactant gas concentrations were measured using a Digilab FTS-20/80 FTIR spectrometer. High-resolution spectral measurements (0.25 $cm^{-1}$) were converted to concentrations using a spectral fitting procedure [17] in conjunction with the HITRAN spectral database [18].

### 2.2. Catalytic experiments

The catalytic measurements were carried out using a single-pass flow system. Reactant mixtures nominally corresponded to 1100 ppm nitromethane with and without 3% $O_2$ and/or 1000 ppm of NO. They were prepared by diluting 10% oxygen/helium and 1% nitric oxide/helium mixtures using Brooks mass flow controllers. The 1100 ppm nitromethane was prepared by passing 10 $cm^3$/min STP of He through a saturator containing nitromethane at ice temperature. Water was introduced in an analogous manner. The combined flow of 100 $cm^3$(STP)/min was passed over ~ 0.1 g samples of Co-ZSM5 contained in a 4 mm i.d. Pyrex U-tube. The section containing the catalyst was mounted inside an aluminium block located at the centre of a tube furnace.

The Co-ZSM5 catalyst (Si/Al ratio 11.2, 4.2 wt% Co, donated by Dr. J. Armor of Air Products and Chemicals), was supplied as a fine powder and had a cobalt content equivalent to a Co/Al ratio of 0.55, implying an exchange level 110% of theoretical. Further details of its preparation can be found elsewhere [2]. For the present experiments, the powder was pressed at 250 MPa (2500 $kg/cm^2$) for 1 min. The resultant discs were crushed and sieved to give a 300–600 $\mu$m particle fraction. The test samples were dried under helium at 110°C overnight and then heated to 500°C (at 5°C/min) in 10% $O_2$/He and held at that temperature for a minimum of 1 h before being cooled to reaction temperature in an atmosphere of flowing helium.

The exit stream of the reactor was periodically sampled into a high-speed micro gas chromatograph (GC) (MTI model M200) and also passed through a 16 cm pathlength infrared cell in the beam of a Mattson Cygnus (model 100) FTIR spectrometer operated at a 0.25 $cm^{-1}$. All tubing between the reactor and the cell, and the cell itself, was heated to 105 ± 5°C using a combination of nichrome wire and heating tape. The GC was configured to give parallel analyses for $O_2$, $N_2$, $CH_4$ and CO (on a molecular sieve 5A column) and $CO_2$, $N_2O$ and $C_2$ hydrocarbons (on a Poraplot U column) with a cycle time of 2 min and a detection limit of < 3 ppm. The FTIR spectrometer was used to determine $CH_3NO_2$, NO, $NO_2$, $NH_3$, HNCO and HCN. Analyses for the first four were based on the intensities of individual lines calibrated against standard mixtures of similar concentrations. HCN was determined by fitting against a spectrum generated using the HITRAN database [17,18] and HNCO using an extinction coefficient estimated in previous work [19].

## 3. Results and discussion

### 3.1. Gas phase experiments

In figure 1 the reactant decomposition profiles for nitromethane oxidation in the presence and absence of NO and $O_2$ are illustrated. Under the conditions of these experiments, it can be seen that nitromethane oxidation occurs above 440°C. In contrast to what has been observed for studies [9] of hydrocarbon oxidation in the presence of NO, the oxidation profiles in the presence and absence of NO are indistinguishable.

In figure 2 the product profiles obtained from the gas phase oxidation and pyrolysis of nitromethane in the presence and absence of NO are presented. As with the study of Tricot et al. [15] CO, $CO_2$, NO, $NO_2$ and water were detected as major products, with traces of formaldehyde also being detected. The CO and $CO_2$ profiles from the oxidation experiments were the same whether NO was added or not. In addition to these products, small quantities of HNCO were also detected in the absence of NO. As the nitrogen-containing products with added NO are difficult to interpret they are omitted from figure 2.

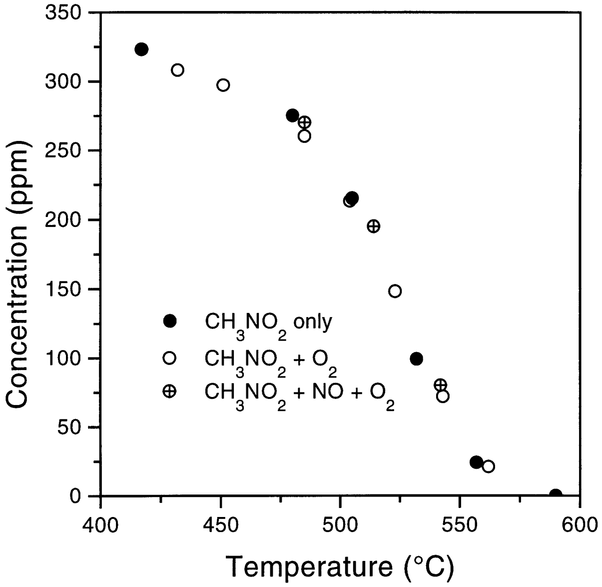

Figure 1. Nitromethane remaining as a function of oxidation/pyrolysis temperature. Initial concentration of $CH_3NO_2$ 330 ppm, residence time 0.4 s at 500°C. (●) No NO or $O_2$ in the feed; (○) 2.95% $O_2$ in the feed; (⊕) 150 ppm NO and 2.95% $O_2$ in the feed.

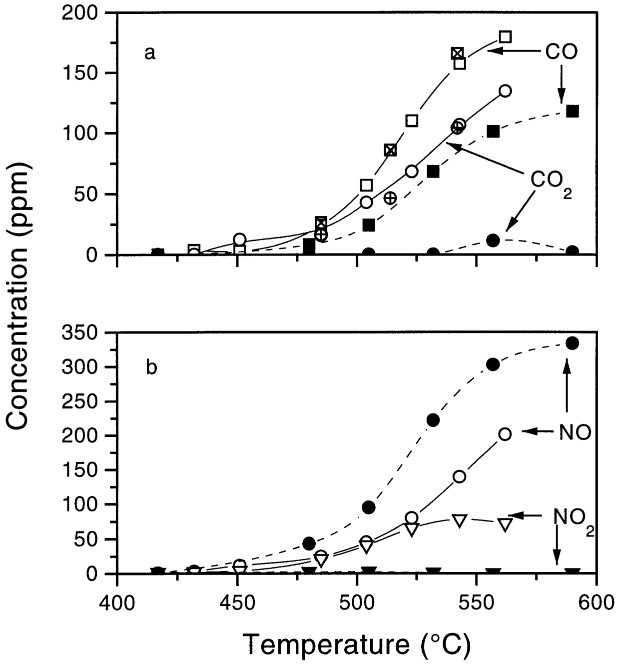

Figure 2. Major products from the oxidation/pyrolysis of nitromethane as a function of temperature. Conditions and feed compositions as for figure 1. (a) Carbon-containing products: (●) $CO_2$ (no $O_2$ in feed), (○) $CO_2$ (with $O_2$ in feed), (⊕) $CO_2$ (with NO and $O_2$ in feed); (■) CO (no $O_2$ in feed), (□) CO (with $O_2$ in feed), (☒) CO (with NO and $O_2$ in feed). (b) Nitrogen-containing products: (●) NO (no $O_2$ in feed), (○) NO (with $O_2$ in feed), (▼) $NO_2$ (no $O_2$ in feed), (▽) $NO_2$ (with $O_2$ in feed).

Carbon and nitrogen mass balances were good (±10%) for the oxidation experiments. In contrast, in the absence of both NO and $O_2$ nitromethane decomposed to form involatile products, resulting in mass balances of less than 60% being obtained at moderate to high extents of decomposition. Under pyrolytic conditions the major products were CO, NO and $H_2O$ (not measured), with no $NO_2$ being detected, and only traces of $CO_2$. HNCO and $CH_2O$ were observed at concentrations slightly higher than experiments where $O_2$ was present. Interestingly, no methane was detected in the products.

Significantly, in the gas phase nitromethane oxidation/pyrolysis experiments, ammonia was not detected as a product. At all temperatures where product measurements were made, the yield of CO exceeded that of $CO_2$, again in contrast to what was observed for the reaction of nitromethane over Co-ZSM-5 (see below).

The results of the gas phase experiments were modelled with the detailed model of methane oxidation in the presence of NO developed previously [9]. The first step in the decomposition is C–N bond scission:

$$CH_3NO_2 \rightarrow CH_3 + NO_2 \qquad (1)$$

followed by reaction between $CH_3$ and $NO_2$:

$$CH_3 + NO_2 \rightarrow CH_3O + NO \qquad (2)$$

Subsequent reactions of the $CH_3O$ result in the formation of formaldehyde, CO and $CO_2$. The rate of the process is largely determined by the rate of reaction (1), in agreement with previous studies of nitromethane decomposition [16]. In the absence of oxygen the only N-containing product observed is NO; no $NO_2$ was detected or predicted. As discussed above, the addition of $O_2$ to the system does not result in a change in the overall rate of nitromethane disappearance but it does change the relative amounts of NO and $NO_2$. With $O_2$ present the following reaction competes with reaction (2) for the methyl radicals produced by C–N bond scission:

$$CH_3 + O_2 \rightarrow CH_3O_2 \qquad (3)$$

and the methylperoxy radicals produced effectively convert the NO to $NO_2$:

$$CH_3O_2 + NO \rightarrow CH_3O + NO_2 \qquad (4)$$

Note that the other product of reaction (4) is $CH_3O$, as for reaction (2) above. This provides an explanation as to why the rate of decomposition of nitromethane is not affected by the addition of $O_2$.

In contrast to many hydrocarbon systems [9] the addition of NO to nitromethane/$O_2$ mixtures also does not result in any changes to the decomposition rate. In the case of the hydrocarbons the promotion of the hydrocarbon oxidation has been attributed to the conversion of an unreactive $HO_2$ species to a more reactive OH by the added NO:

$$HO_2 + NO \rightarrow OH + NO_2 \qquad (5)$$

With nitromethane, reaction (5) does not play an important role, and hence addition of NO does not change the decomposition rate but the relative amounts of NO and $NO_2$ will be affected by the added NO. Hence these products are not included in the results presented in the figures.

### 3.2. *Catalytic experiments*

The temperature dependence for the catalytic decomposition of nitromethane in the presence and absence of oxygen and nitric oxide is shown in figure 3. The data were collected at random with each point representing a quasi-steady state reached after ~ 30 min. As for the homogeneous reaction the concentration profile for nitromethane is similar for experiments carried out with and without $O_2$ and NO. However, as may be seen from figure 4, the product distribution is very different. In the absence of oxygen the major products were $CO_2$ and $NH_3$ in near equimolar amounts. Thus the overall reaction is largely represented by

$$CH_3NO_2 \rightarrow NH_3 + CO_2 \qquad (6)$$

The same is true at temperatures below 340°C with $O_2$ present. At higher temperatures the $NH_3$ concentration falls precipitously to below the detection limit

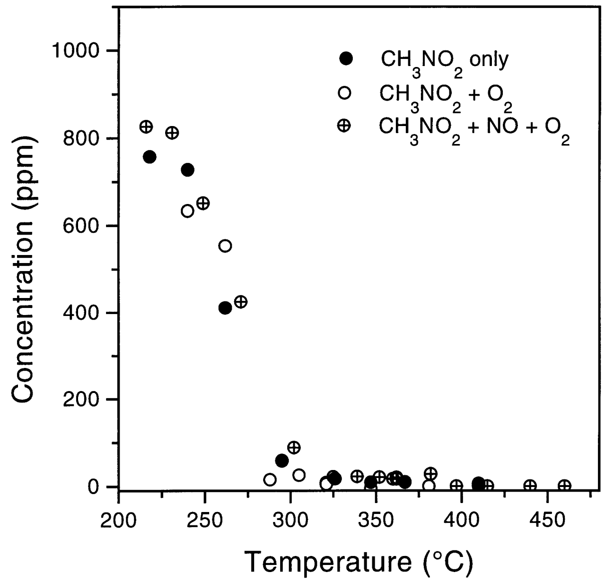

Figure 3. Nitromethane remaining as a function of oxidation/decomposition temperature over 0.1 g of Co-ZSM5. Initial concentration of $CH_3NO_2$ 1100 ppm, flowrate 100 $cm^3$(STP)/min. (●) No NO or $O_2$ in the feed; (○) 2.9% $O_2$ in the feed, (⊕) 1000 ppm NO and 2.9% $O_2$ in the feed.

(< 15 ppm) by 370°C. Nitrogen accounts for more than 60% of the nitrogen in the $CH_3NO_2$ reacted. Nitric oxide comprises most of the rest. Hence the pathway to $N_2$ under these conditions is largely ammonia oxidation

$$4NH_3 + 3O_2 \rightarrow 2N_2 + 6H_2O \qquad (7)$$

It is also apparent from figure 4 that only negligible amounts of $N_2$ are found in the absence of oxygen.

Table 1 shows the effect of including 1000 ppm NO, either alone or in combination with $O_2$, at temperatures above and below the point at which $NH_3$ disappeared. Inclusion of NO has little effect on product yield at 290 and at 430°C in the absence of $O_2$. The same is true when $O_2$ is present at 290°C but at 430°C the yield of $N_2$ is substantially higher than when $O_2$ alone is used. Thus nitric oxide is participating in $NH_3$ removal via the ammonia SCR reaction

$$4NO + 4NH_3 + O_2 \rightarrow 4N_2 + 6H_2O \qquad (8)$$

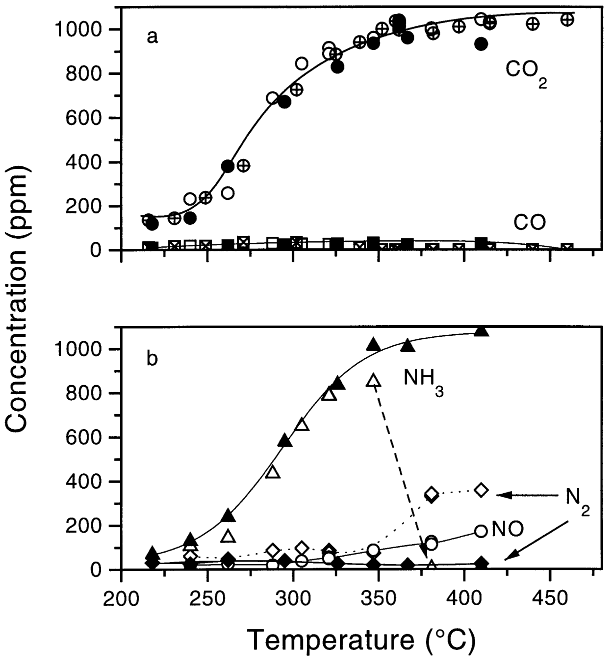

Figure 4. Major products from the catalytic oxidation/pyrolysis of nitromethane over Co-ZSM5 as a function of temperature. Conditions and feed compositions as for figure 3. (a) Carbon-containing products: (●) $CO_2$ (no $O_2$ in feed), (○) $CO_2$ (with $O_2$ in feed), (⊕) $CO_2$ (with NO and $O_2$ in feed); (■) CO (no $O_2$ in feed), (□) CO (with $O_2$ in feed), (☒) CO (with NO and $O_2$ in feed). (b) Nitrogen-containing products: (▲) $NH_3$ (no $O_2$ in feed), (△) $NH_3$ (with $O_2$ in feed); (◆) $N_2$ (no $O_2$ in feed), (◇) $N_2$ (with $O_2$ in feed); (○) NO (with $O_2$ in feed). The amount of NO formed with no $O_2$ in the feed (not shown) was close to the detection limit (~ 20 ppm).

Calculations based on the additional $N_2$ production suggest that this accounts for about one-half of the $NH_3$ removed. It is interesting to note that HCN, which is a significant product at 290°C, disappeared simultaneously with $NH_3$ in the presence of $O_2$.

As noted earlier, the results in figures 3 and 4 were obtained under quasi-steady state conditions which were reached after operation for ~ 30 min at each temperature. However the catalyst was not indefinitely stable at constant temperature when below ~ 320°C. After some hours the conversion of nitromethane commenced to decline steadily with a change in product distribution.

Table 1
The effect of NO on product distribution (in ppm) during decomposition of $CH_3NO_2$ over Co-ZSM5 [a]

| Feed mixture | 290°C | | | | 430°C | | | |
|---|---|---|---|---|---|---|---|---|
| | $N_2$ | $NH_3$ | HCN | $CO_2$ | $N_2$ | $NH_3$ | HCN | $CO_2$ |
| $CH_3NO_2$ | 40 | 580 | 140 | 670 | 40 | 910 | 88 | 930 |
| $CH_3NO_2 + NO$ | n.d. [a] | n.d. | n.d. | n.d. | 75 | 875 | 88 | 920 |
| $CH_3NO_2 + O_2$ | 86 | 434 | 88 | 687 | 370 | < 15 | < 5 | 1030 |
| $CH_3NO_2 + NO + O_2$ | 90 | 584 | 71 | 725 | 560 | < 15 | < 5 | 1110 |

[a] With a feed stream containing 1100 ppm $CH_3NO_2$, and, where present, 1000 ppm NO and/or 2.9% $O_2$ at a total flow rate of 100 $cm^3$(STP)/min over ~ 0.1 g of catalyst.

[b] n.d.: not determined.

Yields of $CO_2$ and $NH_3$ dropped faster than nitromethane conversion. Conversely, and most notably, formation of isocyanic acid HNCO, increased and eventually became the major nitrogen-containing product. Table 2 summarises the result from one test at 290°C. HNCO was below the detection limit (~ 15 ppm) after 100 min but reached 240 ppm, approaching twice that of $NH_3$, after 280 min. Changes in the concentrations of $CO_2$, $N_2$ and HCN showed the same trends as those of $NH_3$. The concentrations of NO, $NO_2$, $N_2O$ and CO were smaller (at most 35 ppm) and relatively more constant.

Introduction of 3000 ppm water after 300 min resulted in conversion of most of the HNCO to $NH_3$ and $CO_2$ and stabilised the activity of the catalyst. A major inference is clear. The conversion of $CH_3NO_2$ to $NH_3 + CO_2$ proceeds in two stages with HNCO as an intermediate

$$CH_3NO_2 \rightarrow HNCO + H_2O \tag{9}$$

$$HNCO + H_2O \rightarrow NH_3 + CO_2 \tag{10}$$

When the catalyst is first placed on stream there are sufficient sites to hydrolyse all HNCO to $NH_3$ and $CO_2$ (figure 3). However, over time at temperatures below 320°C sites are either consumed or blocked, possibly by a polymer of HNCO such as its trimer, cyanuric acid. Hydrolysis then declines and HNCO eventually breaks through as a gaseous product (table 2). Higher temperatures, and/or inclusion of water in the feed, can prevent or delay the deactivation. This interpretation seems reasonable given that cyanuric acid melts and decomposes at the temperature range of interest here and HNCO is readily hydrolysed on alumina [20,21] and Cu-ZSM5 [22]. The mechanism by which HNCO is derived from nitromethane is a matter of speculation. One candidate is dehydration of its enol tautomer, $CH_2{=}NO(OH)$. The natural product would be HCNO but isomerization to produce the thermodynamically more stable HNCO could readily occur via a cyclic intermediate such as that suggested by Blower and Smith [12] for the conversion of nitromethane to ammonia and carbon dioxide over metal-exchanged X and Y zeolites.

The present work may have relevance to the mechanism of the methane/$NO_x$ reaction over Co-ZSM5 and similar catalysts such as Co-ferrierite. The evidence is that its rate-determining step is hydrogen abstraction [4], possibly by adsorbed $NO_2$ [5–7], i.e.

$$CH_4 + NO_2(ads) \rightarrow CH_3 + HONO \tag{11}$$

Nitromethane could then be formed by reaction of the methyl species with $NO_2$,

$$CH_3 + NO_2 \rightarrow CH_3NO_2 \tag{12}$$

At the GHSV used here the methane/$NO_x$ reaction commences at ~ 360°C [2]. The results in figure 1 demonstrate that purely gas phase reactions of nitromethane are unlikely to play a significant role in subsequent reactions at that temperature. However, the data in figure 3 shows that nitromethane would be completely decomposed catalytically. Furthermore, in the presence of oxygen the $NH_3$ formed would be converted to $N_2$ by the oxidation reaction (7) and the ammonia SCR reaction (8) when NO is present as well (table 1). Thus, if $CH_3NO_2$ was formed by reactions (11) and (12) during the methane-SCR reaction over Co-ZSM5 then rapid reaction to give $CO_2$ and $N_2$ via the sequence (9), (10), (7) and/or (8) would seem feasible.

While the scheme discussed above for the methane-SCR reaction appears feasible for Co-ZSM5 there is, as yet, no direct evidence that nitromethane is formed and the possible applicability of the scheme to other systems remains to be established. The $CH_4/NO_x$ reaction is known to proceed over other catalysts, such as Ga-ZSM5 [23,24] and H-ZSM5 [7], which would not be expected to be active for ammonia oxidation or ammonia SCR. In those cases one might expect to see some ammonia in the products or else other pathways would need to be inferred as well. It is interesting to note that ammonia and HNCO have been observed in small amounts as a product of the higher hydrocarbon/$NO_x$/$O_2$ reaction over Cu-ZSM5 [25,26], and there are separate suggestions that this reaction could proceed, at least in part, via ammonia [27,28]. Certainly Cu-ZSM5 is an active ammonia-SCR catalyst [29,30]. There are also suggestions that nitrocompounds and HNCO participate in the propene/$NO_x$/$O_2$ reaction over Ce-ZSM5 [10] and Cu-ZSM5 [11,31] although the emphasis has been on their reaction with $NO_x$ to give nitrogen rather than via hydrolysis to ammonia although that is also feasible [22]. Further work is needed to establish the relevance of the present findings to those systems.

Table 2
Variation in product distribution (in ppm) with time on stream during the reaction of $CH_3NO_2$ with $O_2$ over Co-ZSM5 at 292°C [a]

| Time on stream (min) | $H_2O$ added | Conversion $CH_3NO_2$ (%) | $CO_2$ | $NH_3$ | HNCO | HCN | $N_2$ |
|---|---|---|---|---|---|---|---|
| 100 | nil | 94 | 732 | 485 | < 15 | 96 | 101 |
| 280 | nil | 55 | 221 | 141 | 241 | 35 | 36 |
| 320 | 3000 | 52 | 442 | 367 | 64 | 59 | 52 |

[a] With a feedstream comprising 1100 ppm $CH_3NO_2$, 2.9% $O_2$ in He at a total flow rate of 100 $cm^3$(STP)/min over ~ 0.1 g of catalyst.

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