

Treatment of Effluents from Ammonia Plants – Part III

Ozonation of Amines in an Effluent from a Reforming Plant serving an Ammonia Complex

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Abstract

The process condensate which arises from the operation of an ammonia plant contains methylamines, methanol and ammonia. Oxidation of the methylamines in aqueous solution with ozone was investigated. Trimethylamine in the unionised form reacts most readily with ozone. Good conversion of trimethylamine to the non-odorous oxidation product could be achieved with a 1 : 1,5 ratio of amine to ozone when ozone was passed into a packed tower through which an alkaline solution of the amine was flowing. Dimethylamine and monomethylamine did not react as readily. This is not unexpected; both alkalinity and the influence of CH_3 -groups will affect the reactivity of the $\equiv\text{N}$: group, it follows also that NH_3 in aqueous solution will not react readily and this was confirmed in tests.

The composition of the trade effluent (amines, ammonia, carbon dioxide and methanol) is such that the pH is buffered in the range 8,5-8,7 and effective ozonation of the amines is not possible without addition of a considerable amount of alkali. There is no reaction with methanol.

It is doubtful whether the nuisance value of the effluent can be sufficiently reduced by destruction of trimethylamine mainly, since, although dimethylamine has much less odour than trimethylamine, this odour together with the somewhat pungent, distinct smell of monomethylamine may still be objectionable.

Introduction

A process condensate from the synthesis gas stream of an ammonia plant contains methylamines, methanol, ammonia and dissolved carbon dioxide.

The contaminants can be present to the following extent:

methanol	900-2 000	mg ℓ^{-1}
ammonia	up to 600	mg ℓ^{-1}
methylamines	up to 200	mg ℓ^{-1}
carbon dioxide	saturated	

The total quantity of the process condensate is of the order of 30 $\text{m}^3 \text{h}^{-1}$ and the actual concentration of methylamines depends on the age and condition of the catalysts and operating conditions. A particular sample, for example, gave the following analyses for methylamines:

mono-methylamine	8	mg ℓ^{-1}
di-methylamine	2	mg ℓ^{-1}
and trimethylamine	4	mg ℓ^{-1}

The effluent under consideration tended to be buffered in the range pH 8-8,7 at ambient temperature (approximately 25°C) and it is under alkaline conditions that the fishy odour of the amine is a nuisance to the community.

The obvious treatment to avoid odour is to maintain the effluent acidic, but for various reasons a technique which would destroy or permanently alter the amines to non-odorous compounds was preferred.

As ozone may be produced on site and because of the many advantages it appears to have over other oxidants including chlorine and hydrogen peroxide, preliminary investigations were carried out to determine the effectiveness of ozone in destroying the methylamines, in particular trimethylamine, which is the most offensive of the three methylamines.

General Review

Methylamines are produced by reaction of ammonia and methanol over dehydrating catalysts at 300-500°C and it is not unexpected therefore that amines are found in the condensate involved.

The basicity of the amines compared with that of ammonia is given below (Ginsburg 1967, Patai 1968)

	pKa	pKb
ammonia	9,25	4,75
methylamine	10,63	3,37
di-methylamine	10,78	3,22
tri-methylamine	9,80	4,20

The percentage ionisation of species in solution at different pH values is reflected in Fig 1 which gives the relation for infinite dilution; no correction has been made for ionic strength of the effluent.

The oxidation of amines

Strecker and Thienemann (1920) and Strecker and Baltes (1921) carried out investigations on the action of ozone on ammonia and aliphatic substitution products of ammonia. It was shown that both nitrite and nitrate were formed by action of O_3 on pure liquid ammonia, but that the reaction did not take place as readily in aqueous solution. Pure trimethylamine reacted explosively with ozone, but when diluted with solvents, for example chloroform, trimethylamine oxide was formed. Reactions with monomethylamine and dimethylamine were also investigated.

In work by Ross (1964) it was shown that the conjugate acid of trimethylamine, R_3NH^+ is much less susceptible to oxidation than trimethylamine itself.

Bailey *et al* (1968) in a series of papers concerning reactions of ozone with amines, indicated that these reactions all involve an initial electrophilic attack of ozone on the nitrogen to give an adduct for which different routes are available:

- loss of molecular oxygen to give an amine oxide or further reaction products thereof
- an intra molecular oxidation of a side chain, and dissociation to nitrogen cation radicals and an ozonate anion radical, followed by further reactions of these.

It should, however, be noted that the predominant path will vary with the oxidant, the amine and the solvent. Similarly the rate of reaction will be affected and Bailey (1973) for example, has indicated that electrophilic attack of ozone on the nucleophilic nitrogen of amines would be expected to be slow in aqueous medium due to solvation.

By analogy with reactions reported in the literature, reactions of the following nature could be expected when using methylamines:

Trimethylamine

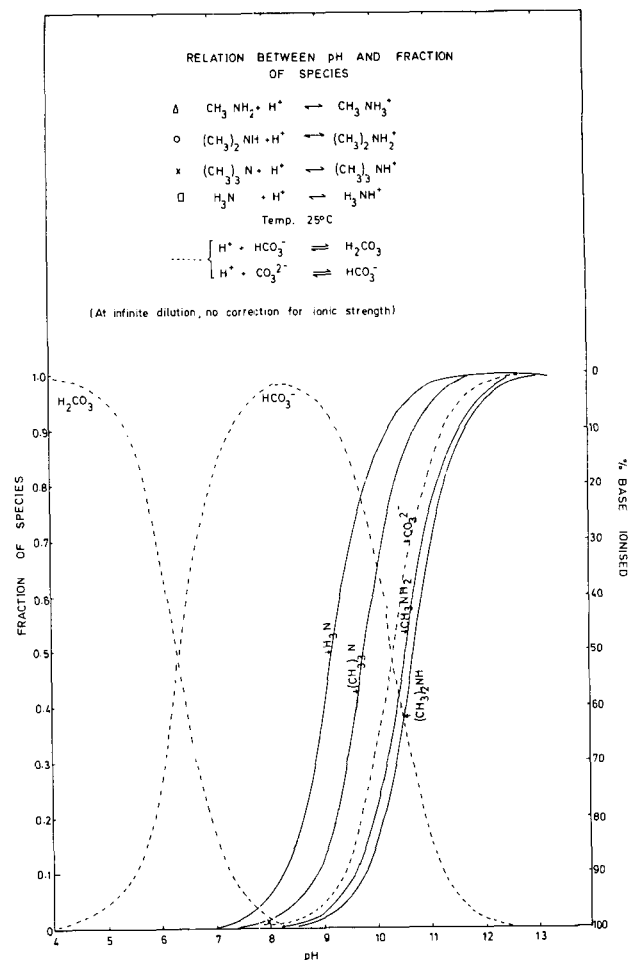
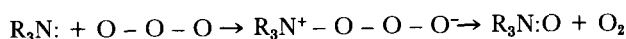
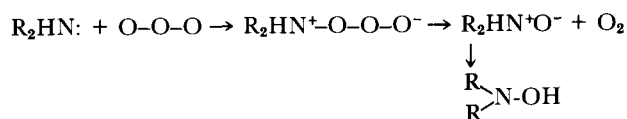
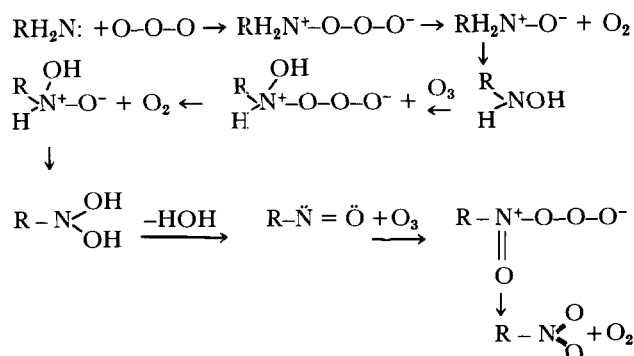


Figure 1
Relation between pH and fraction of species

Dimethylamine



Monomethylamine



In the above, $R = CH_3$

The mechanism of amine oxide formation has a first order dependence on both amine and ozone concentrations and most certainly involves a slow electrophilic attack by the terminal oxygen on the amine lone pair electrons shown by the fact that the conjugate acid of the amine is unreactive.

In the pollution abatement field Rogozhkin (1970) has reported on the removal of dimethylamine from waste waters by ozonation. It is claimed that ozonation of waste waters containing dimethylamine should be carried out in alkaline medium. The products of oxidation are reported as formaldehyde, formic acid, carbonic acid, nitrites and nitrates. However the specific consumption of ozone was high.

Hewes and Davidson (1971) in their article on kinetics of ozone decomposition and reaction with organics in water, mention the reaction between di-isopropylamine and ozone with special reference to the attack in the amine group compared with that on a C-C bond.

With regard to the attack of ozone on ammonia, Papko (1957) showed that ammonia is easily oxidised by ozone to nitrates, but that the reaction is somewhat slow in water solution. Singer and Zilli (1975) conducted investigations on the ozonation reaction in waste water. They concluded that the oxidation reaction is first order in respect of the concentration of ammonia and is catalysed by OH^- over the pH range 7-9. Again the molar ratio of ozone consumed per ammonia oxidised was high. In work on the oxidation of ammonia in the gas phase however, Olszyna and Heicklen (1972) showed at ammonia/ozone ratios of from 5-12, when ammonia was added to ozone, a white aerosol, which deposited on the walls of the reactor, was formed almost immediately. For concentrations of ozone greater than 10 Torr, however, an explosion resulted when the ammonia was added.

A considerable amount of information on the chemistry and use of ozone is documented in the American Chemical Society Advances in Chemistry Series No. 21, 1959, No. 77, 1968 and No. 112, 1972 and in the field of pollution abatement Evans (1972) has edited a series of papers concerning ozone, its manufacture, use and reactions with some groupings. The Environmental Protection Agency, USA, has also issued some reports on ozone oxidation.

Decomposition of ozone

In the use of ozone in aqueous media, the decomposition of ozone in water undoubtedly leads to other reactive species which may be more favourable for the oxidation of certain materials (Peleg 1976). This theme is developed by Hoigne (1976) and Hoigne and Bader (1976) who have shown that ozone may either react directly with substrates or, above a critical pH value, decompose before reacting. It is beyond this critical pH value that decomposition products of ozone such as hydroxyl radicals (OH^\bullet) become the important oxidants.

Ozone production and commercial ozonisers

On an industrial scale, ozone is produced by passing very dry atmospheric air (or oxygen) through a high voltage electric discharge. The concentration of ozone obtained is proportional to the intensity of discharge and is between 10-30 g ozone/m³ air at 20°C with an optimum at 12-15 g/m³.

Since ozonation is a rapidly emerging technology, there are now a number of reliable and relatively low cost generators available from a number of manufacturers.

Hazards involved in bulk storage of powerful oxidants are eliminated with ozone since the gas is produced as required.

Experimental

Ozone

Ozone was produced from a small laboratory ozoniser and from a commercial generator (Model LG-2-L2 W.R. Grace & Co.). Dry air was used as the oxygen source in both generators.

Analytical procedures

Ozone was analysed by reaction with potassium iodide and titration of released iodine by sodium thiosulphate, based on the methods described by Birdsall *et al* (1952), and by Ingols *et al* (1959).

Methylamines and alcohol were measured by gas chromatography and total carbon by a Beckman Total Organic Carbon Analyser.

Nitrates were measured using an Orion specific ion nitrate electrode.

Nitrite was determined spectrophotometrically using the α -naphthylamine, sulphanilic acid reaction.

Reaction of methylamines with ozone – batch operation

Preliminary tests

Initial tests were conducted on trimethylamine solution and a solution consisting of trimethylamine, methanol and ammonia. In these tests 100 ml of solution in a gas washing bottle was treated with ozone generated in a laboratory ozonizer.

Each 100 ml was ozonized for a definite time period and then analysed for residual TMA.

The results of these tests are given in Figure 2.

It will be seen that the reaction follows first order kinetics (with respect to TMA) very closely over most of the reaction. During the course of the reaction the pH decreased significantly and the presence of ammonia and methanol had no material effect on the course of the reaction. The molar ratio of amine destroyed to ozone used was high in the first few minutes (approximately 1).

Similar results were obtained with dimethylamine. The results of tests with dimethylamine are given in Figure 3.

Batch tests were carried out in the apparatus shown in Figure 4, where the pH could be controlled by the addition of sodium hydroxide during ozonation. Samples for analysis were withdrawn from the apparatus at fixed intervals.

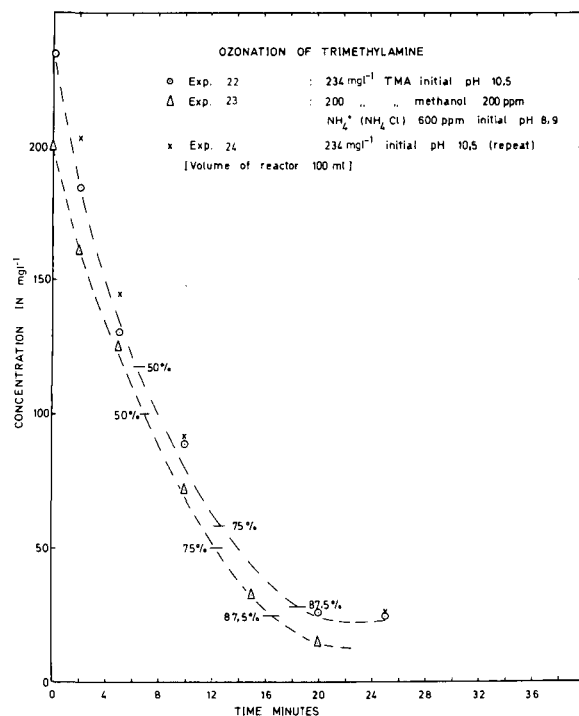


Figure 2
Ozonation of trimethylamine

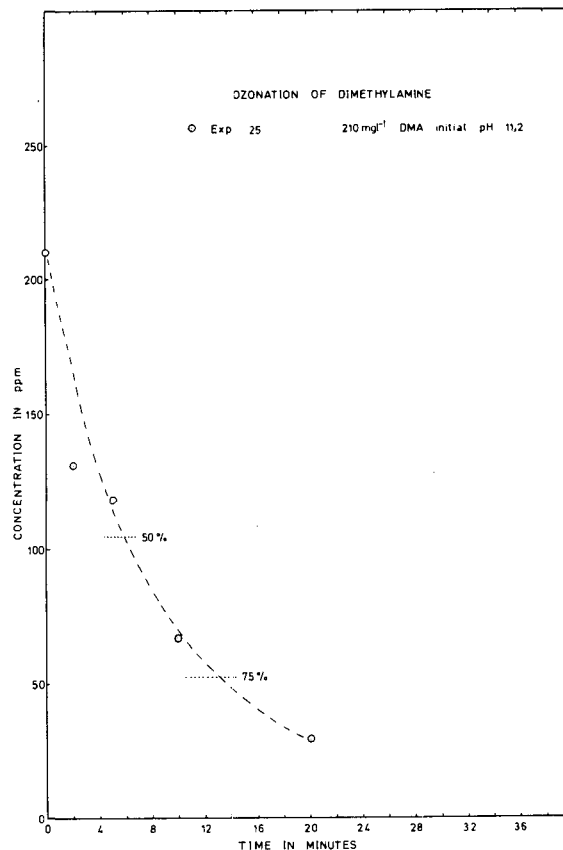


Figure 3
Ozonation of dimethylamine

FIGURE 3
TEST: 25
AMINE CONCENTRATION mg l^{-1} : DMA 210

mg O ₃ per 100 ml reactor liquid/ min	Time	Molar ratio amine destr ozone used	pH	Gas flow ml min ⁻¹	O ₃ in mg min ⁻¹	O ₃ in mg l ⁻¹	O ₃ % v/v	O ₃ in mg	O ₃ out mg	O ₃ reacted %	NO ₂ formed mg l ⁻¹	NO ₃ formed mg l ⁻¹
	0		11,2									
2,1	2	1,18	11,1	121	2,1	17,4	0,81	4,2	0,12	97	0,19	<1
2,14	5	0,95	11,1	110	2,14	19,5		10,7	nil	100	0,27	<1
2,02	10	0,75	10,4	135	2,02	15,0		20,2	0,3	98,5	0,47	2
—	15	—	—	—	—	—		—	—	—	—	—
2,02	20	0,48	8,5	135	2,02	15,0		40,4	0,6	98,5	0,45	6,4

Ozone generated from a laboratory ozoniser.
Volume of amine solution in reactor 100 ml.
pH not controlled during ozonation.

The results of tests on mono, di-, and trimethylamine are given in Figures 5 and 6.

In these tests the quantity of ozone was such that ozone was always present in the exit gas.

The first order disappearance rate of amine applies in the case of dimethylamine and trimethylamine at the higher pH values.

A group of tests on dimethylamine at pH values ranging

from 8-10 are reflected in Figure 7. The effect of pH is evident in all tests.

A sample of the trade effluent was treated with excess ozone in the apparatus shown in Figure 4. No adjustment of pH was undertaken because of the strong buffer effect at pH 8,5-8,7 because of the carbonate content in the effluent. Even at this pH trimethylamine followed a first order disappearance rate, dimethylamine reacted in the pattern shown in Figure 7, for pH 8-9 and monomethylamine reacted more slowly but in the pattern shown in Figure 5.

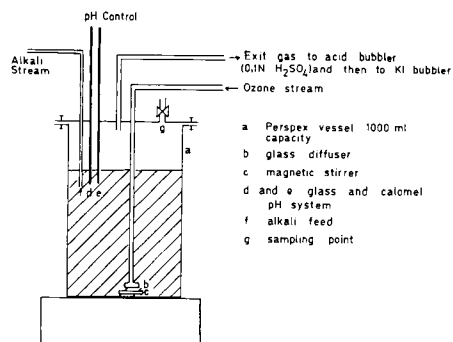


Figure 4
Apparatus used for reaction of ozone with amines

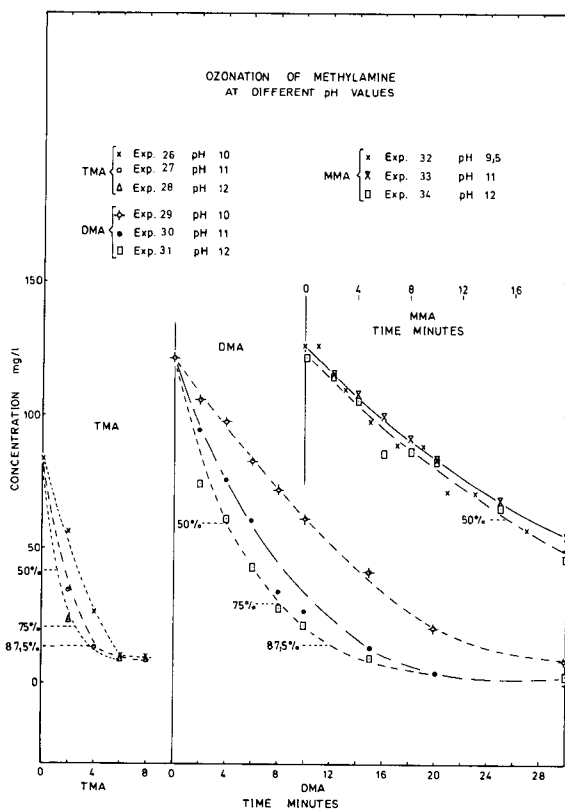


Figure 5
Ozonation of methylamine at different pH values

FIGURE 5

mg O ₃ per 100 ml reactor liquid/ min	Test	Amine conc. mg l ⁻¹	pH control- led	Gas Flow l min ⁻¹	O ₃ In mg min ⁻¹	O ₃ mg l ⁻¹	O ₃ %
TMA							
11,3	26 x	84	10	6,5	68	10,5	0,53
11,3	27 o	84	11	6,5	68	10,5	0,53
11,3	28 Δ	84	12	6,5	68	10,5	0,53
DMA							
11,3	20 ↗	121,5	10	6,5	68	10,5	0,53
11,3	30 ●	122	11	6,5	68	10,5	0,53
11,3	31 □	120	12	6,5	68	10,5	0,53
MMA							
10,7-12	32 x	126	9,5	8	64-72	9,8-9	,44-45
10,5	33 X	125	11	5,5	63	11,5	0,58
11,3	34 □	121	12	6,5	68	10,5	0,53

Ozone generated from a commercial generator.
Volume of amine solution in reactor = 600 ml.
Ozone in excess immediately after start of experiments.

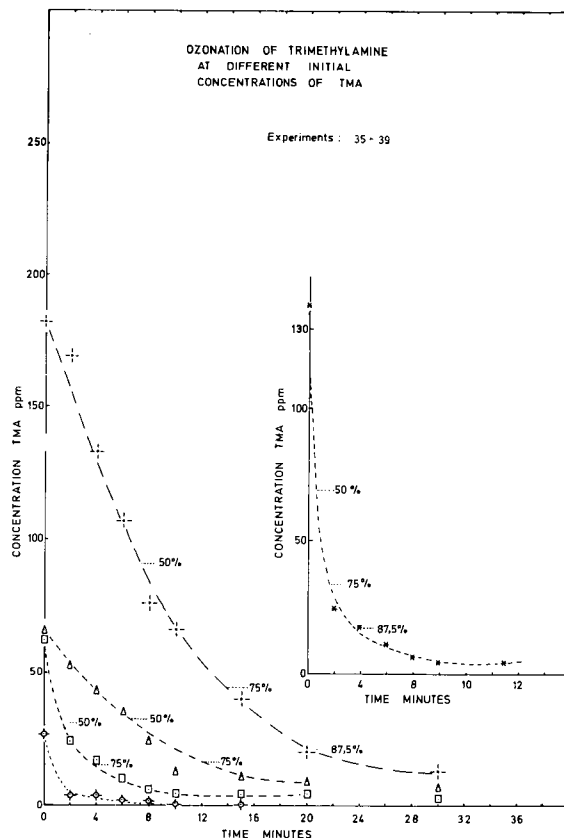


Figure 6
Ozonation of trimethylamine at different initial concentrations of TMA

FIGURE 6

mg O ₃ per 100 ml reactor liquid/ min	Test	TMA conc.	pH (control- led)	Gas flow	O ₃ in	O ₃	O ₃ %	
		mg ℓ ⁻¹		ℓ min ⁻¹	mg min ⁻¹	mg ℓ ⁻¹	v/v	
2,13	Δ	35	66	9,5	1,6	12,8	8,0	0,4
2,13	↗	36	182	9,5	1,6	12,8	8,0	0,4
10,3	□	37	62	9,8	5,5	61,6	11,2	0,56
10,3	*	38	138	9,8	5,5	61,6	11,2	0,56
10,3	↗	39	27	9,5	5,5	61,6	11,2	0,56

Ozone generated from a commercial ozoniser.
Volume of amine solution in reactor, 600 ml.
Ozone in excess immediately after start of experiments.
Temperature of tests: ambient 22-25°C.

To determine the extent of oxidation of ammonia, a solution containing NH₄Cl was ozonised at pH 8, 9 and 10. For a feed, containing about 470 mg l⁻¹ NH₃-N only 8, 10 and 11 mg l⁻¹ NO₃-N and 0,015, 0,01 and 0,005 mg l⁻¹ NO₂-N were formed at the respective pH values. Similar tests on an aqueous solution of methanol (2000 ppm) gave an insignificant reduction.

Possible stripping of amine during ozonation

When air alone is passed through solutions of amines or ammonia at high pH values stripping of the components will occur as may be expected from data given in Figure 1.

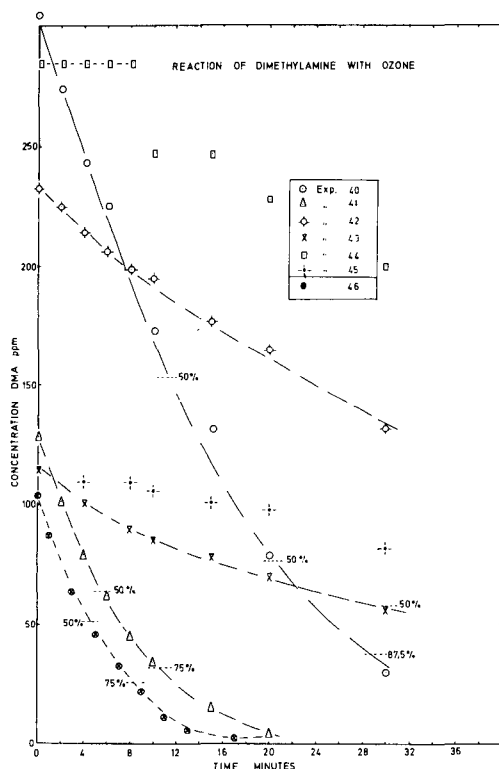


Figure 7
Reaction of dimethylamine with ozone

FIGURE 7

mg O ₃ per 100 ml reactor liquid per min	Test	DMA conc.	pH control- led	Gas flow	O ₃ in	O ₃	O ₃ %	Temperature	
		mg ℓ ⁻¹		ℓ min ⁻¹	mg min ⁻¹	mg ℓ ⁻¹	v/v	°C	
10,5	⊙	40	304	10	5,5	63	11,5	0,58	ambient
10,5	Δ	41	128	10	5,5	63	11,5	0,58	ambient
10,5	+	42	233	9	5,5	63	11,5	0,58	ambient
10,5	×	43	116	9	5,5	63	11,5	0,58	ambient
10,5	□	44	285	8	5,5	63	11,5	0,58	ambient
10,5	⬆	45	113	8	5,5	63	11,5	0,58	ambient
11,7–12,0	⊗	46	103,5	9,5	8,0	70–72	8,8–9	0,44–0,45	42,5–44,5°C

Ozone generated from a commercial ozoniser.

Volume of amine solution in reactor, 600 ml.

Ozone in excess immediately after start of experiments.

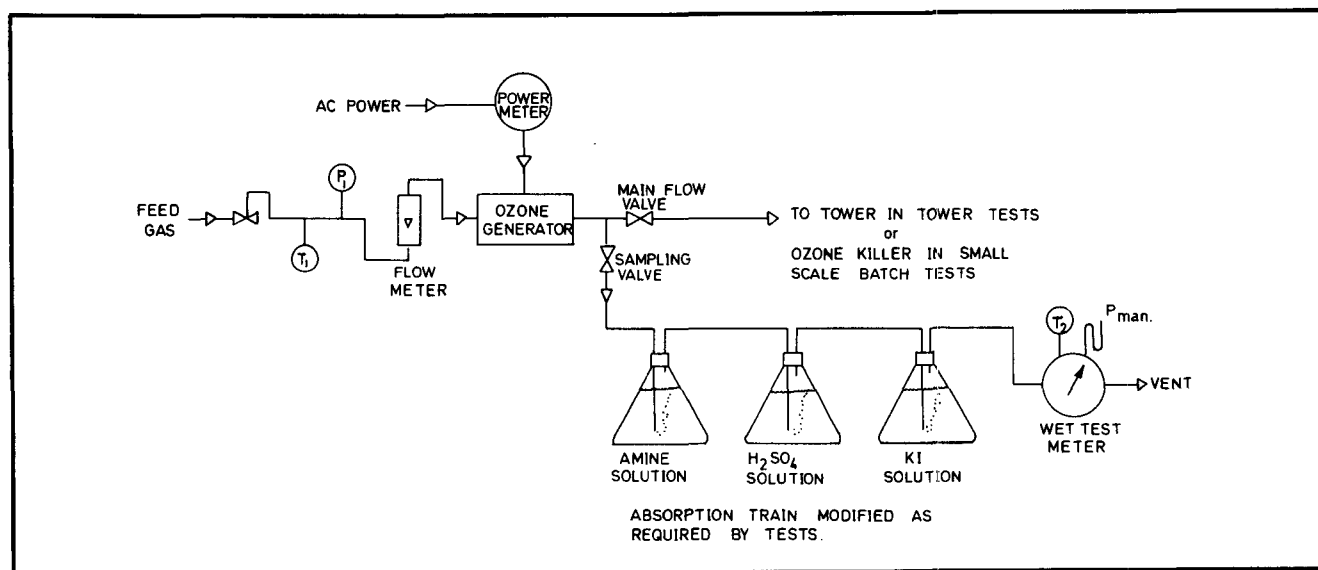


Figure 8
Schematic flow production and use of ozone

When air containing ozone is passed through solutions of amines at high pH values, the extent of loss is expected to be low because of chemical reaction.

A few tests of a semi-quantitative nature were conducted in an apparatus similar to that shown in Figure 8. Air/ozone mixtures at the rate of 1,5 l min⁻¹ equivalent to 12 mg ozone min⁻¹ were passed through 100 ml of amine solution. In the case

of trimethylamine, at an initial pH of 10,3, 5% of the trimethylamine was left in the reactor after 5 minutes, but over 90% equivalent total organic carbon was still present in the reactor. Only 5% of the amine was stripped and trapped in the acid bubbler. Similar findings were obtained with regard to total organic carbon left in the reactor after ozonation of dimethylamine and monomethylamine, at initial pH values of 10,8. In the case of dimethylamine, 5% DMA was left in the

reactor, but over 86% equivalent total organic carbon. In the case of monomethylamine, the corresponding figures were 50% MMA and 84% equivalent total organic carbon.

When ozone is reacted with the gaseous forms of the amines including ammonia, white fumes are formed which settle out on the walls of the reaction vessels. The reaction is immediate with TMA and the white aerosol copious. This same effect can be observed in the larger bubbles rising through a reaction vessel when treating amines in solution with ozone gas.

Continuous operation

Because of limitations in time, it was necessary to proceed with tests involving continuous ozonation of methylamine contaminated solutions.

Tests were conducted in a packed tower (as shown in Figure 9). The tower was packed with 1 cm x 1 cm ceramic Raschig rings.

Since the reaction with amines exhibited first order kinetics with respect to amine concentration it would be desirable to treat feed solutions with low concentrations of amines. In the tests conducted, a concentration range of 10-200 mg ℓ^{-1} was used.

In view of the marked effect of pH on the course of the reactions, caustic soda was metered into the tower either

entering at the top or middle zone of the tower so as to control the pH to a particular value at the outlet of the tower.

Using a mol ratio of O_3 /amine of 1,06, the conditions for the packed tower column were derived from 'Technical data related to tower packing' Norton Chemical Process Division, 1970.

The results of tests are given in Tables 1 and 2. In all cases an attempt was made to achieve maximum destruction of amine with little or no ozone in the exit gas.

Trimethylamine tests

Comparison of tests 1 and 3; 4, 5, and 2 shows the marked effect of pH on the reaction, not only in regard to amine destroyed, but also in regard to ozone consumption.

At a ratio of 1,5 ozone to amine, all the amine (except for a small amount lost by stripping) was destroyed. This is also evident in test 6 where the ratio of ozone to amine was 1,24 and a reasonable destruction of amine was achieved even though the outlet pH of the liquid was only 8,7. The different flow patterns of liquid and gases in tests 11-13 did little to improve the reaction.

For trimethylamine then, satisfactory destruction of the amine can be expected with an inlet pH of about 9,8 controlled to an out-let of about 9,4 with an aminé to ozone ratio of 1,5.

Dimethylamine and monomethylamine tests

Results of tests with DMA (test 15 and 16) were not as promising, but indications are that at pH values above 10,5 and at ozone : amine ratios higher than used here, reasonable destruction could be achieved.

The position with mono-methylamine (tests 17 and 18) is as expected. Even at very high pH values and excess ozone, the reaction is slow.

Trade effluent tests

Tests 19, 20 and 21 were conducted on the actual effluent from the factory. The high carbonate content buffered the effluent to pH 8,5-8,7 and a large addition of caustic soda would have been required to increase the pH to suitable levels.

Tests were thus run at pH values of 8,9 and 10,6 only. Results were disappointing and without further work on the interaction of the various components in the effluent an ozonation process cannot at this stage be recommended for treatment of the effluent.

Discussion and Conclusions

The course of the reactions is not unexpected. An examination of Figure 1, showing the relation between pH and species ionised, shows that at pH 9 for trimethylamine the ratio

$(CH_3)_3NH^+/(CH_3)_3N$ is of the order of 6,3;

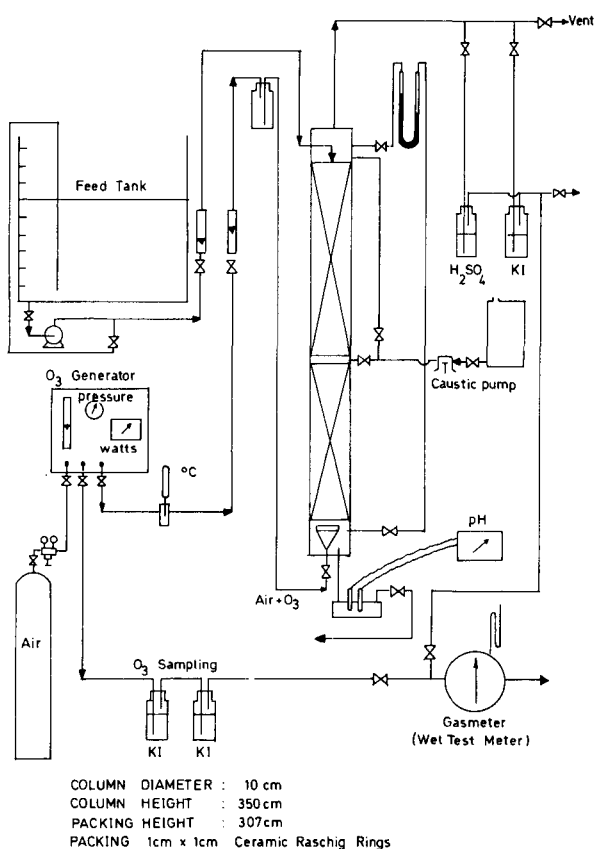


Figure 9
 Pilot plant set up (schematically)

TABLE 1
OZONISATION OF AMINES IN PACKED TOWER (Counter current operation ambient temperature)

Exp. No.	Liquid flow $\ell\ m^{-1}$	Gas flow $\ell\ m^{-1}$	$L \left[\frac{\rho_G}{\rho_L - \rho_G} \right]$	0,5	Amine feed mm ℓ^{-1}	pH in	pH out	Inlet ozone mm ℓ^{-1}	Exit ozone mm ℓ^{-1}	Amine in mol h^{-1}	Amine out mol h^{-1}	Amine in gas exit mol h^{-1}	Amine destr. mol h^{-1}	Ozone in mol h^{-1}	Ozone out mol h^{-1}	Ozone consumed mol h^{-1}	Tot. O ₂ Total amine	Amine destr. Ozone summed	Amine destr. %	Ozone summed %	Amine
1.*	4,2	21	5,8		0,681	9,0	6,9	0,148	0,081	0,172	0,103	—	0,069	0,186	0,103	0,083	1,08	0,83	40,1	44,6	TMA
2.	4,2	21	5,8		0,822	9,1	9,4	0,176	0,013	0,207	0,029	0,003	0,175	0,221	0,016	0,025	1,07	0,85	84,5	92,7	TMA
3.*	4,2	21	5,8		0,707	10,1	9,7	0,152	—	0,178	0,038	0,001	0,139	0,192	—	0,192	1,08	0,72	78,1	100	TMA
4.	4,2	21	5,8		0,720	10,1	10,0	0,162	—	0,181	0,031	0,002	0,148	0,204	—	0,204	1,13	0,73	81,8	100	TMA
5.	4,2	21	5,8		0,720	10,1	10,0	0,161	—	0,181	0,031	0,002	0,148	0,202	—	0,202	1,12	0,73	81,7	100	TMA
6.*	4,2	21	5,8		0,702	10,0	8,7	0,174	0,007	0,177	0,029	—	0,148	0,219	0,009	0,210	1,24	0,70	83,6	95,9	TMA
7.	4,2	21	5,8		0,707	10,1	10,0	0,212	0,005	0,178	—	negl.	0,178	0,267	0,007	0,260	1,5	0,68	99,8	97,5	TMA
8.*	4,2	21	5,8		0,683	11,0	10,5	0,188	0,003	0,172	—	0,002	0,170	0,237	0,004	0,233	1,38	0,73	98,8	98,3	TMA
9.	4,2	21	5,8		0,686	9,0	10,0	0,204	0,026	0,173	0,011	0,001	0,161	0,237	0,032	0,225	1,49	0,72	93,1	87,6	TMA
10.	4,2	21	5,8		0,344	9,0	10,0	0,087	0,012	0,087	0,009	negl.	0,078	0,109	0,016	0,093	1,25	0,84	89,7	85,3	TMA
11.	3,2	23,0	4,03		1,271	9,5	9,4	0,240	—	0,244	0,029	negl.	0,215	0,331	—	0,331	1,36	0,65	88	100	TMA
12.	3,8	55,3* ¹	2,0		1,722	9,7	9,4	0,157	0,002	0,393	0,028	negl.	0,365	0,520	0,005	0,515	1,32	0,71	93	99	TMA
13.	2,0	67* ¹	0,87		3,339	9,8	9,2	0,136	0,0016	0,401	0,073	negl.	0,328	0,448	0,005	0,443	1,12	0,74	82	99	TMA
14.	4,2	21	5,8		0,618	9,8	9,4	0,194	0,026	0,156	—	—	0,156	0,244	0,033	0,211	1,56	0,74	100	86,5	TMA

*1 Beyond capacity of generator so ozone ex generator was supplemented with air before entry into tower.

*pH not controlled during experiment.

**mm = millimole

TABLE 2
OZONISATION OF AMINES IN PACKED TOWER (Counter current operation)

Exp. No.	Liquid flow $\ell\ m^{-1}$	Gas flow $\ell\ m^{-1}$	$\frac{L}{G} \left[\frac{\rho_G}{\rho_L - \rho_G} \right]$	0,5	Amine feed mm ℓ^{-1}	pH In	pH Out	Inlet Ozone mm ℓ^{-1}	Exit Ozone mm ℓ^{-1}	Amine In mol h^{-1}	Amine Out mol h^{-1}	Amine in Gas exit mol h^{-1}	Amine destr. mol h^{-1}	Ozone In mol h^{-1}	Ozone Out mol h^{-1}	Ozone Consum.	Tot. O ₂ Total amine	Amine destr. O ₂ Consum.	Amine destr. %	Ozone consum. %	Amine	temp.		
15.	4,2	21	5,8		0,500	10,1	10,0	0,123	0,008	0,126	0,060	0,001	0,065	0,154	0,010	0,144	1,22	0,45	51,6	93,5	DMA	ambien		
16.	4,2	21	5,8		0,387	10,5	10,5	0,107	0,005	0,097	0,028	0,001	0,068	0,135	0,006	0,129	1,39	0,53	70,1	95,6	DMA	ambien		
17.	4,2	21	5,8		0,309	9,95	10,0	0,181	0,059	0,078	0,042	0,0008	0,035	0,228	0,074	0,154	2,92	0,23	44,8	67,5	MMA	ambien		
18.	4,2	21	5,8		0,403	10,0	10,5	0,180	0,042	0,102	0,051	0,0009	0,050	0,227	0,053	0,174	2,23	0,29	48,7	76,6	MMA	ambien		
19.	4,2	21	5,8	{	Tri	8,9	8,8	0,210	0,005	Tri	Tri	Tri	Tri	0,265	0,006	0,259	0,84	0,25	Tri	37,5	97,5	Trade effluent	28°C	
					0,254					0,0641	0,0401	0,0002	0,024						Di					
					0,269					0,0678	0,0486	negl.	0,0192						Di					
					Mono					0,0678	0,0486	negl.	0,0192						Mono					
20.	4,2	21	5,8	{	0,722	10,5	10,4	0,233	—	0,1819	0,1593	negl.	0,0226	0,293	—	0,293	0,92	0,36	Tri	62,6	100	Trade effluent	43°C	
					Tri					Tri	Tri	Tri	Tri						Di					
					0,264					0,0666	0,0240	0,0004	0,0414						Di					
					0,246					0,0620	0,0408	negl.	0,0212						Mono					
21.	4,2	21	5,8	{	Mono	10,5	10,2	0,232	—	0,1888	0,1472	negl.	0,0416	0,292	—	0,292	0,90	—	Tri	55,6	100	Trade effluent	62°C	
					0,749					Tri	Tri	Tri	Tri						Tri					
					Tri					Tri	Tri	Tri	Tri						Tri					
					0,215					0,054	0,021	0,003	0,030						Tri					
D & M*					D & M*					D & M*					D & M					negl.				

* Combined MMA + DMA in = 30,8 ppm out = 22,6 ppm

*1 Sample received contained 960 ppm MeOH, 12-15 ppm TMA, 12 ppm DMA, 23 ppm MMA.