

# Identification of corrosion inhibiting long-chain primary alkyl amines by gas chromatography and gas chromatography–mass spectrometry

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## Abstract

Gas chromatography with flame-ionization detection (FID) and gas chromatography–mass spectrometry (GC/MS) has been used to identify long-chain primary alkyl amines after derivatization with trifluoroacetic anhydride (TFAA). Electron impact ionization (EI) and positive chemical ionization (PCI) mass spectra of trifluoroacetylated derivatives of the identified alkyl amines are presented.

The corrosion inhibiting alkyl amines were applied in the investigation of a new anticorrosive and antifouling formulation for water–steam circuit of energy systems in the power industry.

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**Keywords:** GC-FID; GC/MS; Primary long-chain alkyl amines; Corrosion inhibitors

## 1. Introduction

The determination of amines is of great importance to the chemical and pharmaceutical industry as well as for environmental analysis [1]. Amines are widely used as materials or intermediates in the production of polymers, plastics and coating additives, dyes, pharmaceuticals, ion-exchange resins, pesticides, petroleum products additives, and more recently as corrosion inhibitors in chemical treatment of boiler water in water–steam systems of power plants. Traditionally, the most common way to reduce corrosion is to add a corrosion inhibitor to a corrosive system to preserve metals. Corrosion inhibitors are chemical compounds or formulations of these which, when present in small amounts in an aggressive medium, inhibit corrosion by bringing about changes in the surface condition of the metal [2–5]. They should be low toxic, biodegradable and soluble in aqueous medium. One of the largest classes of organic

corrosion inhibitors is the organic amine class. Aliphatic and aromatic amines, mono-, di- or polyamines and their salts, all are used as corrosion inhibitors [6]. Applying the ideas of green chemistry to the class of organic amines corrosion inhibitors has resulted in reduced use of many of the more toxic aromatic amines/salts and the introduction of long-chain aliphatic mono-, di- or polyamines or their salts as replacement [6].

In our previous paper [7], we have reported on a sensitive gas chromatographic method for the identification and quantitative determination of trifluoroacetylated derivatives of commercially available *tert*-octadecylamines of Primene JM-T<sup>TM</sup>-type in water samples of boiler water, superheated steam and condensate from the power plant. The corrosion inhibiting alkyl amines (Fig. 1) were applied in a new technology of boiler water chemical treatment in energy systems in the power industry [2,3,8–14]. The objective of the present work is the separation and identification of corrosion inhibiting long-chain primary alkyl amines by gas chromatography and gas chromatography–mass spectrometry. The EI- and PCI-mass spectra of trifluoroacetylated derivatives of these alkyl amines

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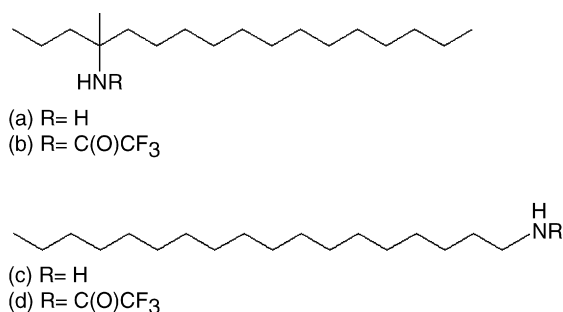


Fig. 1. Chemical structures of selected investigated primary long-chain alkyl amines and their trifluoroacetylated (TFA) derivatives: (a) 4-methyl-4-heptadecaneamine; (b) *N*-(4-methyl-4-heptadecyl)-trifluoroacetamide; (c) 1-amino-octadecane; (d) *N*-(1-octadecyl)-trifluoroacetamide.

Table 1  
Retention data of trifluoroacetylated (TFA) derivatives of *tert*-octadecylamines in Primene JM-T<sup>TM</sup> (Fig. 1)

Peak	Capillary column						Proposed chemical structure
	DB-5ms			DB-17ms			
	$t_R^a$ (min)	$I_p^b$	$\Delta I_p^c$	$t_R^a$ (min)	$I_p^b$	$\Delta I_p^c$	
A	34.17	1971	–	27.06	1987	–	9-Methyl-9-heptadecaneamine-TFA
B	34.21	1972	1	27.12	1989	2	8-Methyl-8-heptadecaneamine-TFA
C	34.32	1975	3	27.28	1994	5	7-Methyl-7-heptadecaneamine-TFA
D	34.53	1981	6	27.54	2002	8	6-Methyl-6-heptadecaneamine-TFA
E	34.85	1990	9	28.01	2017	15	5-Methyl-5-heptadecaneamine-TFA
F	35.50	2009	19	28.80	2042	25	4-Methyl-4-heptadecaneamine-TFA
G	36.62	2048	39	30.35	2091	49	3-Methyl-3-heptadecaneamine-TFA
H	36.96	2052	4	30.48	2096	5	2-Methyl-2-heptadecaneamine-TFA

<sup>a</sup>  $t_R$ : retention time.

<sup>b</sup>  $I_p$ : retention index in temperature programmed GLC [22,23].

<sup>c</sup>  $\Delta I_p$ : increment of retention indices  $I_{p(n+1)} - I_{p(n)}$ .

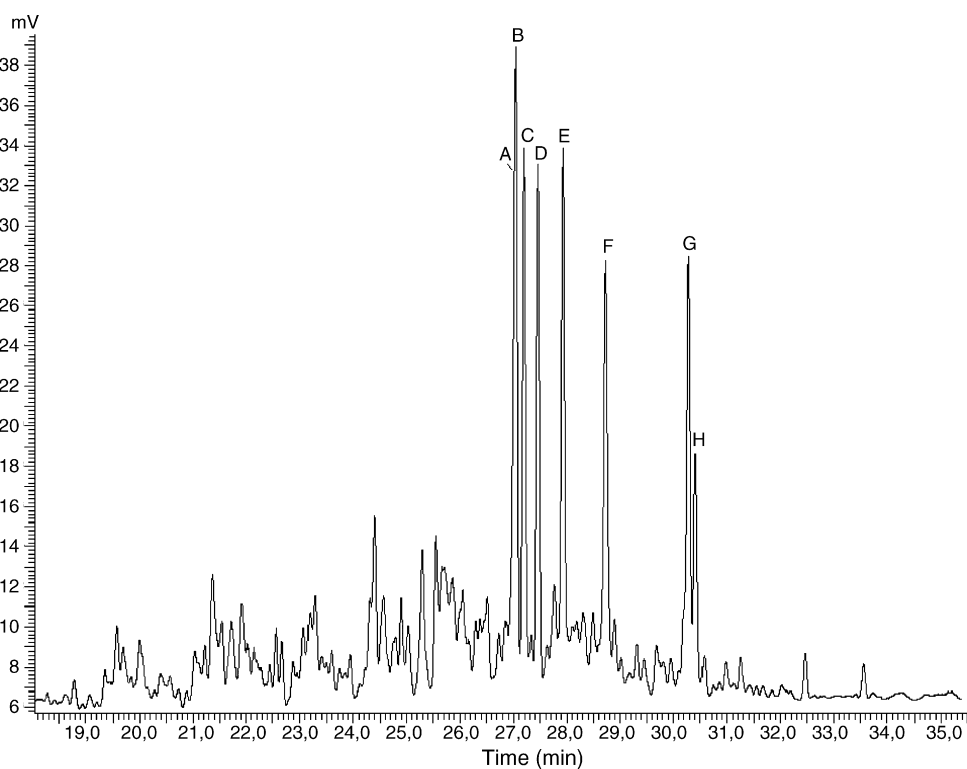


Fig. 2. GC/FID chromatogram of trifluoroacetylated (TFA) derivative of Primene JM-T<sup>TM</sup> dissolved in *n*-hexane. Fused silica capillary column: DB-17ms, 60 m  $\times$  0.25 mm i.d., film thickness 0.25  $\mu$ m. Column temperature programmed from 150  $^{\circ}$ C (1 min hold) at 3  $^{\circ}$ C min<sup>-1</sup> to 280  $^{\circ}$ C (hold 30 min). Split/splitless injector: 290  $^{\circ}$ C. Helium constant pressure 120 kPa, split flow 20 cm<sup>3</sup> min<sup>-1</sup>. FID 320  $^{\circ}$ C. For retention data and peak identification, see Table 1.

are reported and discussed for the first time. These results are part of the further investigation of a new class of anticorrosive and antifouling formulation for water–steam circuit of energy systems in the power industry [15]. They are important to understand the processes during its technical use and its ecological and economical impact.

## 2. Experimental

### 2.1. Chemicals

Alkyl amines Primene JM-T<sup>TM</sup> obtained from Rohm and Haas France S.A.S. (Valbonne, France) and Armeen<sup>®</sup> HTD

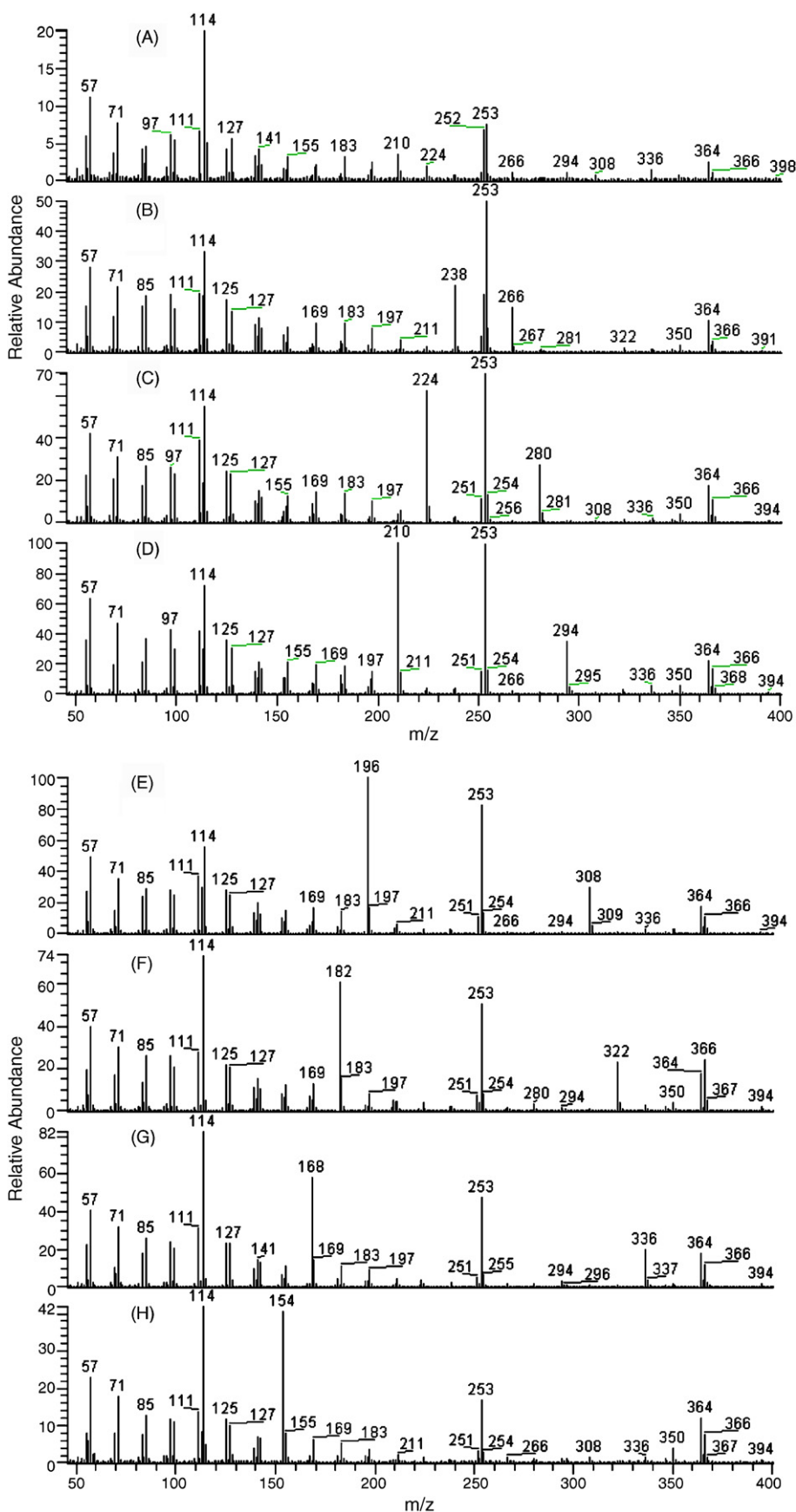


Fig. 3. Positive chemical ionization (PCI) mass spectra of trifluoroacetylated (TFA) derivatives of *tert*-octadecylamines identified in Primene JM-T<sup>TM</sup>. For mass spectra identification, see Table 2.

obtained from AKZO Nobel Surface Chemistry (Arnheim, The Netherlands) as well as 1-hexadecylamine (90%) from ACROS ORGANICS (Geel, Belgium) were used in this investigation.

*n*-Hexane for HPLC (Biosolve B.V., Valkenswaard, The Netherlands), tetrahydrofuran (THF) LiChrosolv for HPLC (Merck), trifluoroacetic anhydride (TFAA) from Macherey-Nagel (Düren, Germany) and 5% dimethyldichlorosilane (DMDCS) in toluene (Sylon CT) from Supelco (Bellefonte, PA, U.S.A.) were used. A mixture of C<sub>6</sub>–C<sub>44</sub> hydrocarbons in cyclohexane (ASTM D2887 Quantitative Calibration Mix) obtained from Supelco was used to calculate the retention index of each compound.

## 2.2. Derivatization

A compact ultrasonic bath Sonorex Super RK 31H from Bandelin electronic (Berlin, Germany) and reaction vessels of 5 ml with solid cap and PTFE liner (Supelco) deactivated with 5% DMDCS in toluene (Sylon CT) were used for derivatization [16]. The derivatization procedure was the same as in our previous work [7].

## 2.3. Instrumentation

Gas chromatographic analyses were performed using an Clarus 500 gas chromatograph from Perkin-Elmer Instru-

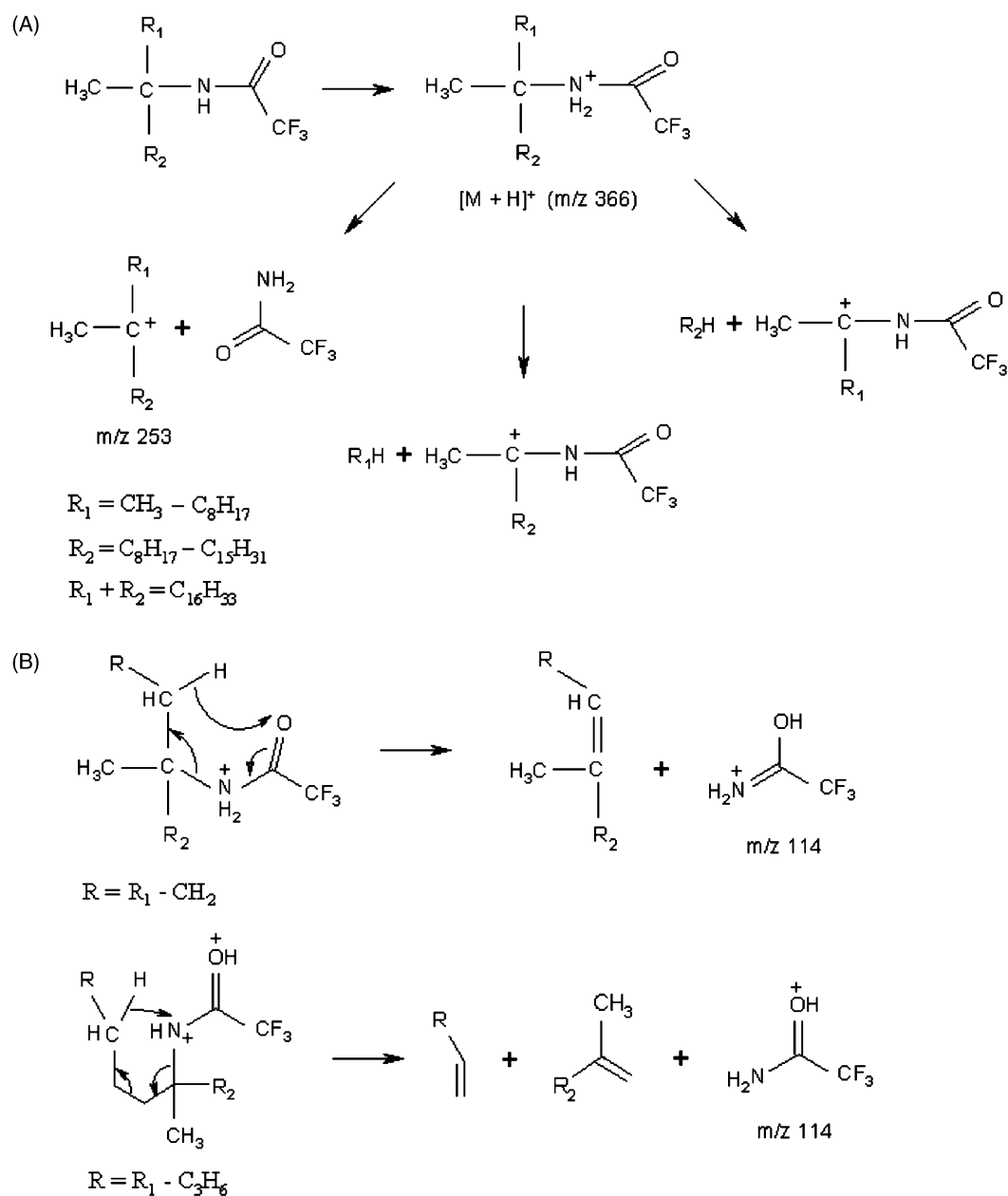


Fig. 4. Proposed PCI-MS fragmentation pattern for investigated *N*-(*tert*-octadecyl)-trifluoroacetamides. (A) cleavage mechanism and (B) McLafferty rearrangement. For mass spectra, see Fig. 3.

Table 2  
Significant fragments and identification of trifluoroacetylated (TFA) derivatives of *tert*-octadecylamines in Primene JM-T<sup>TM</sup> (Fig. 3)

Mass spectrum	Significant fragments ( <i>m/z</i> ) PCI	Proposed chemical structure
A	[M+H-C <sub>8</sub> H <sub>18</sub> ] <sup>+</sup> (252); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	9-Methyl-9-heptadecaneamine-TFA
B	[M+H-C <sub>9</sub> H <sub>20</sub> ] <sup>+</sup> (238); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>7</sub> H <sub>16</sub> ] <sup>+</sup> (266); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	8-Methyl-8-heptadecaneamine-TFA
C	[M+H-C <sub>10</sub> H <sub>22</sub> ] <sup>+</sup> (224); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>6</sub> H <sub>14</sub> ] <sup>+</sup> (280); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	7-Methyl-7-heptadecaneamine-TFA
D	[M+H-C <sub>11</sub> H <sub>24</sub> ] <sup>+</sup> (210); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>5</sub> H <sub>12</sub> ] <sup>+</sup> (294); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	6-Methyl-6-heptadecaneamine-TFA
E	[M+H-C <sub>12</sub> H <sub>26</sub> ] <sup>+</sup> (196); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>4</sub> H <sub>10</sub> ] <sup>+</sup> (308); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	5-Methyl-5-heptadecaneamine-TFA
F	[M+H-C <sub>13</sub> H <sub>28</sub> ] <sup>+</sup> (182); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>3</sub> H <sub>8</sub> ] <sup>+</sup> (322); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	4-Methyl-4-heptadecaneamine-TFA
G	[M+H-C <sub>14</sub> H <sub>30</sub> ] <sup>+</sup> (168); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-C <sub>2</sub> H <sub>6</sub> ] <sup>+</sup> (336); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	3-Methyl-3-heptadecaneamine-TFA
H	[M+H-C <sub>15</sub> H <sub>32</sub> ] <sup>+</sup> (154); [M+H-CF <sub>3</sub> C(O)NH <sub>2</sub> ] <sup>+</sup> (253); [M+H-CH <sub>4</sub> ] <sup>+</sup> (350); [M-H] <sup>+</sup> (364); [M+H] <sup>+</sup> (366)	2-Methyl-2-heptadecaneamine-TFA

Table 3  
Retention data of trifluoroacetylated (TFA) derivative of Armeen<sup>®</sup> HTD (hydrogenated tallow amine) (Fig. 5)

Peak	Capillary column						Proposed chemical structure
	DB-5ms			DB-17ms			
	<i>t<sub>R</sub></i> <sup>a</sup> (min)	<i>I<sub>p</sub></i> <sup>b</sup>	$\Delta I_p$ <sup>c</sup>	<i>t<sub>R</sub></i> <sup>a</sup> (min)	<i>I<sub>p</sub></i> <sup>b</sup>	$\Delta I_p$ <sup>c</sup>	
A	29.70	1843	–	25.67	1943	–	1-Aminotetradecane-TFA
B	33.14	1941	98	28.81	2042	99	1-Aminopentadecane-TFA
C	36.78	2046	105	32.22	2150	108	1-Aminohexadecane-TFA
D	39.94	2139	93	35.10	2241	91	1-Aminoheptadecane-TFA
E	43.54	2245	106	38.50	2349	108	1-Aminooctadecane-TFA
F	46.48	2331	86	41.07	2431	82	1-Aminononadecane-TFA
G	49.92	2424	93	43.96	2525	94	1-Amineicosane-TFA

<sup>a</sup> *t<sub>R</sub>*: retention time.

<sup>b</sup> *I<sub>p</sub>*: retention index in temperature programmed GLC [22,23].

<sup>c</sup>  $\Delta I_p$ : increment of retention indices  $I_{p(n+1)} - I_{p(n)}$ .

ments (Norwalk, CT, U.S.A.), equipped with two split/splitless injectors at 290 °C and two flame-ionization detectors (FIDs) operated at 320 °C. Helium 5.0 grade (Westfalen AG, Münster, Germany) was used as a carrier gas. The helium inlet pressure was 120 kPa and the split flow was 20 cm<sup>3</sup> min<sup>-1</sup>. The fused silica capillary columns used in this investigation were 60 m × 0.25 mm i.d., film thickness 0.25 μm DB-5ms and 60 m × 0.25 mm i.d., film thickness 0.25 μm DB-17ms, both from J&W Scientific. The oven temperature was programmed from 150 °C (1 min hold) at 3 °C min<sup>-1</sup> to 280 °C (hold 50 min). Chromatographic data were processed with TotalChrom Workstation version 6.3 software (Perkin-Elmer Instruments).

GC/MS measurements were made using an ThermoQuest Trace 2000 gas chromatograph (ThermoQuest CE Instruments, Milan, Italy) interfaced to a ThermoQuest/Finnigan Voyager quadrupole mass spectrometer (ThermoQuest/Finnigan Mass-Lab Group, Manchester UK) operated in electron impact ionization (EI) mode and positive chemical ionization (PCI) mode with an ThermoQuest Xcalibur data system, the *NIST 02* mass spectra library, and a CombiPAL autosampler (CTC Analytics AG, Zwingen, Switzerland). The fused silica capillary column, 60 m long, 0.25 mm i.d. with DB-5ms (J&W) stationary phase, film thickness 0.25 μm was used. The tem-

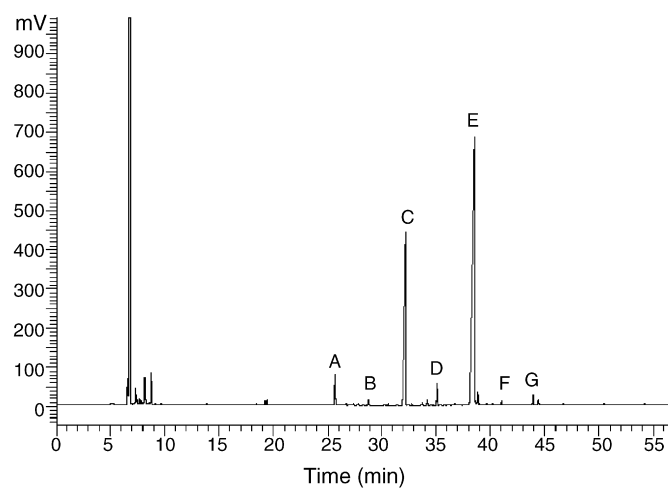


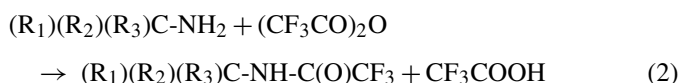
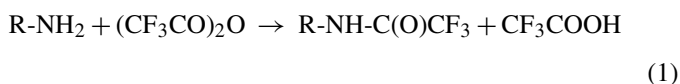
Fig. 5. GC/FID chromatogram of trifluoroacetylated (TFA) derivative of Armeen<sup>®</sup> HTD (hydrogenated tallow amine) in THF. Fused silica capillary column: DB-17ms, 60 m × 0.25 mm i.d., film thickness 0.25 μm. Column temperature programmed from 150 °C (1 min hold) at 3 °C min<sup>-1</sup> to 280 °C (hold 50 min). Split/splitless injector: 290 °C. Helium constant pressure 120 kPa, split flow 20 cm<sup>3</sup> min<sup>-1</sup>. FID 320 °C. For retention data and peak identification, see Table 3.

perature of column was programmed from 60 °C (1 min hold) at 3 °C min<sup>-1</sup> to 280 °C (30 min hold) or from 150 °C (1 min hold) at 3 °C min<sup>-1</sup> to 280 °C (50 min hold). Helium 5.0 grade (Westfalen AG) was used as a carrier gas. Constant flow of helium of 1 cm<sup>3</sup> min<sup>-1</sup> was used during the whole analysis. The temperature of the split/splitless injector was 250 °C and the split flow was 100 cm<sup>3</sup> min<sup>-1</sup>. The transfer line temperature was 280 °C. The EI ion source temperature was kept at 250 °C. The ionization occurred with a kinetic energy of the impacting electrons of 70 eV. The emission current was 250 μA. The detector voltage was 350 V. Methane grade 4.5 (Westfalen AG) was used as reagent gas for PCI. The positive chemical ionization occurred with a kinetic energy of 70 eV. The emission current was 150 μA. The PCI ion source temperature was 250 °C. Mass spectra and reconstructed chromatograms (total ion current [TIC]) were obtained after eluting of solvent (9 min) by automatic scanning in the mass range *m/z* 35–750.

### 3. Results and discussion

Amines are generally known to be very difficult to analyze by gas chromatography due to their basic character [1,7]. In addition to the basic character, the amino group introduces a large dipole in the molecule. This dipole is responsible for strong interaction with silanol groups and siloxane bridges in the structure of the stationary phase of the GC capillary column. This

often results in non-linear adsorption effects and can be seen as strong tailing peaks in the chromatogram. Most critical are the primary alkyl amines and alkyl diamines [17]. The best way to prevent interaction of the strong dipole and to improve the chromatographic separation and the detection sensitivity is a prior derivatization step. Good results from our previous work [7] with acylation of alkyl amines with trifluoroacetic anhydride (TFAA), stimulated us to proceed our work with derivatization of the investigated long-chain alkyl amines (Fig. 1) with the same derivatizing reagent (TFAA) [7,18]:



#### 3.1. Identification of Primene JM-T™

For chromatographic separation of trifluoroacetylated (TFA) derivative of Primene JM-T™ two types of capillary columns with different polarity were tested. The retention data of the investigated TFA derivatives of *tert*-octadecylamines obtained for both capillary columns are summarized in Table 1. As can be seen from Table 1, the increments of retention indices for each

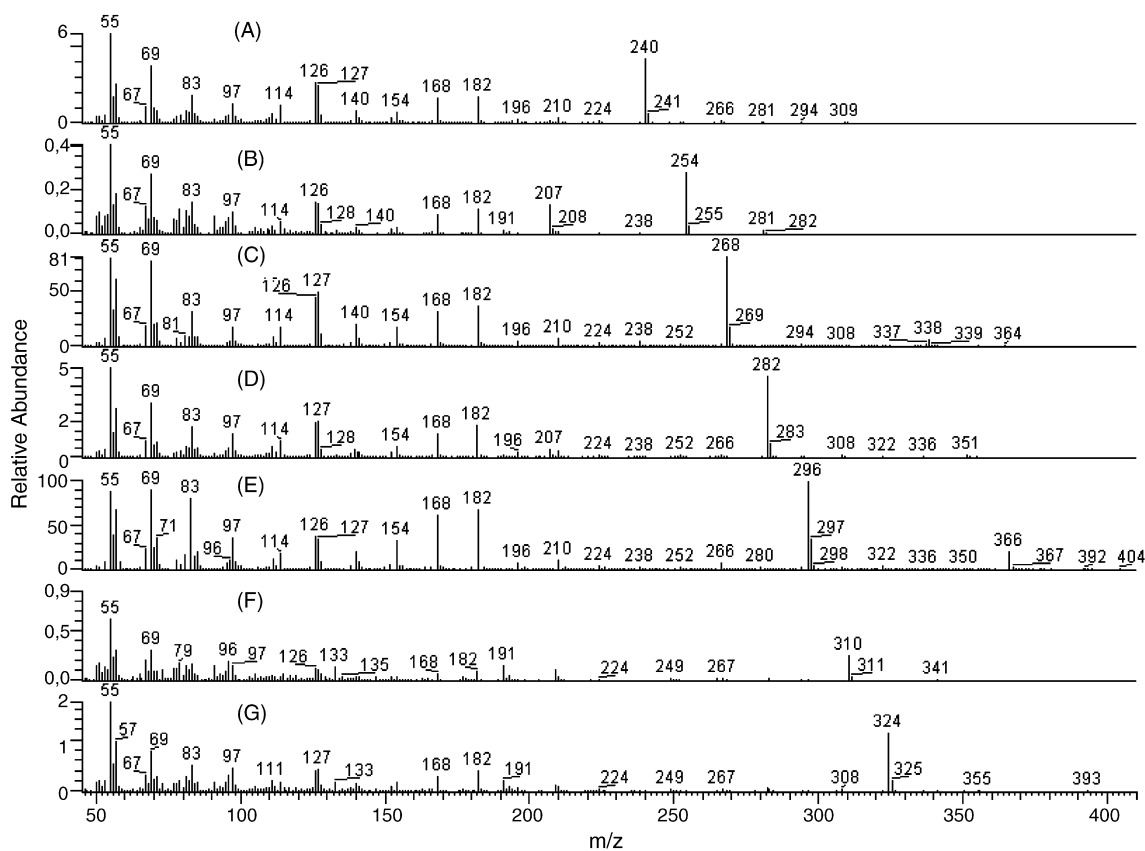


Fig. 6. Electron impact ionization (EI) mass spectra of *n*-alkylamines identified in hydrogenated tallow amine Armeen® HTD. Mass spectra identification: (A) 1-aminotetradecane-TFA; (B) 1-aminopentadecane-TFA; (C) 1-aminohexadecane-TFA; (D) 1-aminoheptadecane-TFA; (E) 1-aminooctadecane-TFA; (F) 1-aminononadecane-TFA; (G) 1-aminoeicosane-TFA.

substance are greater by using moderately polar DB-17ms fused silica capillary column. Thereby, the separation of compounds is better than by using non-polar DB-5ms fused silica capillary column. Fig. 2 shows the chromatogram of trifluoroacetylated derivative of Primene JM-T™ obtained with DB-17ms capillary column. Fig. 3 shows the recorded mass spectra of the identified TFA derivatives of *tert*-octadecylamines. The compounds were detected in the positive chemical ionization mode (PCI) by their quasimolecular ions  $[M+H]^+$  ( $m/z$  366),  $[M-H]^+$  ( $m/z$  364), and by appropriate fragments (Table 2). All mass spectra in Fig. 3 exhibit characteristic alkyl ions:  $[C_4H_9]^+$  ( $m/z$  57),  $[C_5H_{11}]^+$  ( $m/z$  71),  $[C_6H_{13}]^+$  ( $m/z$  85),  $[C_7H_{15}]^+$  ( $m/z$  99),  $[C_8H_{17}]^+$  ( $m/z$  113),  $[C_9H_{19}]^+$  ( $m/z$  127),  $[C_{10}H_{21}]^+$  ( $m/z$  141),  $[C_{11}H_{23}]^+$  ( $m/z$  155),  $[C_{12}H_{25}]^+$  ( $m/z$  169),  $[C_{13}H_{27}]^+$  ( $m/z$  183),  $[C_{14}H_{29}]^+$  ( $m/z$  197) and  $[C_{15}H_{31}]^+$  ( $m/z$  211) differing by 14 u, formed through a cleavage mechanism of the alkyl chain of the branched alkyl amine TFA-derivatives. The characteristic fragments at  $m/z$  253 in Fig. 3 corresponds to the  $(R_1)(R_2)(CH_3)C^+$  carbocations, where  $R_1 = CH_3-C_8H_{17}$ ,  $R_2 = C_8H_{17}-C_{15}H_{31}$  and  $R_1 + R_2 = C_{16}H_{33}$ , formed through a

cleavage mechanism of the quasimolecular ions  $[M+1]^+$  (Fig. 4). Other characteristic fragments in Fig. 3 at  $m/z$  114 are formed in the McLafferty type-reaction of alkene elimination from the *N*-(*tert*-octadecyl)-trifluoroacetamide ions (Fig. 4). The McLafferty rearrangement is common for carbonyl compounds such as ketones, carboxylic acids and esters or amides [19]. The proposed PCI fragmentation pattern for *N*-(*tert*-octadecyl)-trifluoroacetamide is shown in Fig. 4. The proposed chemical structures of the investigated *tert*-octadecylamines are summarized in Tables 1 and 2.

### 3.2. Identification of Armeen® HTD

Fig. 5 shows the GC/FID chromatogram of trifluoroacetylated derivative of Armeen® HTD (hydrogenated tallow amine [20]) in THF obtained by using moderately polar DB-17ms fused silica capillary column. The identification of compounds was carried out by calculation of increments of retention indices and interpretation of the recorded mass spectra of trifluoroacetylated derivatives of Armeen® HTD and 1-hexadecylamine (standard

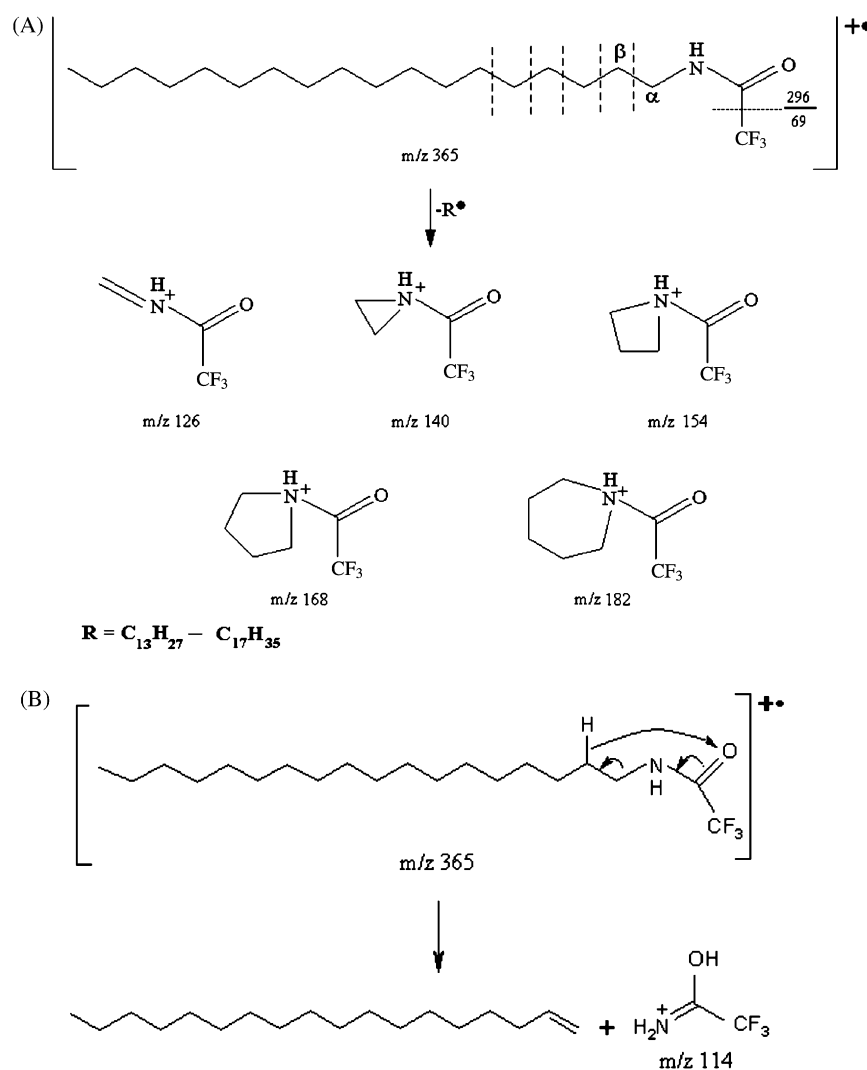


Fig. 7. Proposed EI-MS fragmentation pattern for *N*-(1-octadecyl)-trifluoroacetamide. (A) cleavage mechanism and (B) McLafferty rearrangement. For mass spectra, see Fig. 6.

substance). As can be seen from Table 3, the increments of retention indices of compounds differ for both non-polar and moderately polar capillary columns by average 97 index units. According to basis of the retention index system itself, the theoretical value is equal 100 index units, while the members of a homologous series differ by 100 index units per methylene group [21].

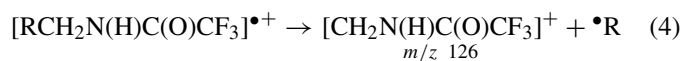
The mass spectra formed by the electron-impact ionization (EI) of the trifluoroacetylated *n*-alkyl amines ( $C_{14}$ – $C_{20}$ ) identified in Armeen<sup>®</sup> HTD are presented in Fig. 6. The characteristic fragments at  $m/z$  240, 254, 268, 282, 296, 310 and 324 in the mass spectra (Fig. 6A–G) are formed by the loss of the  $CF_3$ -radical of 1-aminotetradecane-TFA, 1-aminopentadecane-TFA, 1-aminohexadecane-TFA, 1-aminoheptadecane-TFA, 1-aminooctadecane-TFA, 1-aminononadecane-TFA and 1-aminoeicosane-TFA ions, respectively (3):



where  $R = C_{13}H_{27}$ – $C_{19}H_{39}$ .

The peaks at  $m/z$  241, 255, 269, 283, 297, 311 and 325 (Fig. 6A–G) are probably due to the suitable  $[M+H-CF_3]^+$  ions. The peak at  $m/z$  69 represents the  $[CF_3]^+$  ion. The mass spectra (Fig. 6A, C and E) exhibits also a series of fragments at

$m/z$  126, 140, 154, 168, 182, 196, 210, 224, 238, 252, 266, 280 and 294. They are formed in the alkyl cleavage mechanism of 1-aminotetradecane-TFA, 1-aminohexadecane-TFA and 1-aminooctadecane-TFA, respectively, with a ring formation reaction, producing a series of  $[(H_2C)_nNHC(O)CF_3]^+$  ( $n = 2$ – $13$ ) fragments with the positively charged nitrogen, and abstracting an alkyl radical. The fragment at  $m/z$  126 corresponds to the  $[(H_2C)NHC(O)CF_3]^+$  ion from the  $\beta$ -cleave of the TFA derivatives of the alkylamines (4):



where  $R = C_{13}H_{27}$ – $C_{19}H_{39}$ .

Other characteristic fragments in Fig. 6:  $[C_4H_7]^+$  ( $m/z$  55),  $[C_5H_9]^+$  ( $m/z$  69),  $[C_6H_{11}]^+$  ( $m/z$  83),  $[C_7H_{13}]^+$  ( $m/z$  97) and  $[C_8H_{15}]^+$  ( $m/z$  111) are formed in the McLafferty type-reaction of alkene elimination from the *N*-(alkyl)-trifluoroacetamide ions. The peak at  $m/z = 114$  is due to the  $[F_3CC(OH)NH_2]^+$  ion. The proposed EI-MS fragmentation pattern for *N*-(1-octadecyl)-trifluoroacetamide is shown in Fig. 7.

Fig. 8 shows the mass spectra obtained by the positive chemical ionization (PCI) of the trifluoroacetylated *n*-alkyl amines ( $C_{14}$ – $C_{20}$ ) identified in Armeen<sup>®</sup> HTD. The quasimolecular ions  $[M+H]^+$  ( $m/z$  310, 324, 338, 352, 366, 380 and 394) in Fig. 8A–G

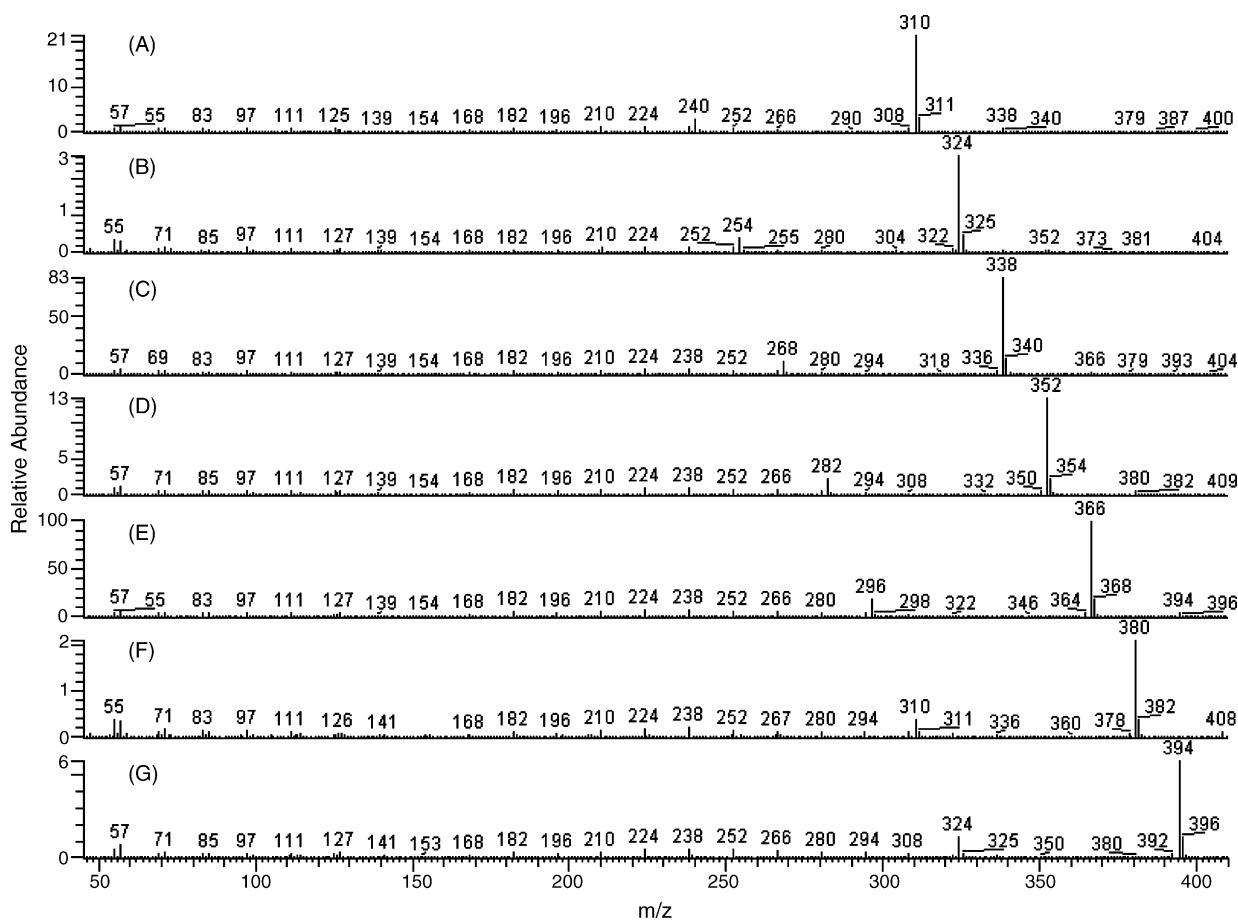


Fig. 8. Positive chemical ionization (PCI) mass spectra of *n*-alkylamines identified in hydrogenated tallow amine Armeen<sup>®</sup> HTD. Mass spectra identification: (A) 1-aminotetradecane-TFA; (B) 1-aminopentadecane-TFA; (C) 1-aminohexadecane-TFA; (D) 1-aminoheptadecane-TFA; (E) 1-aminooctadecane-TFA; (F) 1-aminononadecane-TFA; (G) 1-aminoeicosane-TFA.



corresponds to 1-aminotetradecane-TFA, 1-aminopentadecane-TFA, 1-aminohexadecane-TFA, 1-aminoheptadecane-TFA, 1-aminooctadecane-TFA, 1-aminononadecane-TFA and 1-aminoeicosane-TFA, respectively. The characteristic fragments at  $m/z$  240, 254, 268, 282, 296, 310 and 324 in the mass spectra (Fig. 8A–G) are formed, as in the case of electron-impact ionization of Armeen<sup>®</sup> HTD, by the loss of the CF<sub>3</sub>-radical of 1-aminotetradecane-TFA, 1-aminopentadecane-TFA, 1-aminohexadecane-TFA, 1-aminoheptadecane-TFA, 1-aminooctadecane-TFA, 1-aminononadecane-TFA and 1-aminoeicosane-TFA, respectively. The proposed chemical structures of the investigated *n*-alkyl amines are summarized in Table 3.

#### 4. Conclusion

Gas chromatography with flame-ionization detection and gas chromatography–mass spectrometry were successfully used for separation and identification of commercially available long-chain primary alkyl amines. The identified corrosion inhibiting alkyl amines were applied in the investigation of a new anti-corrosive and antifouling formulation for water–steam circuit of energy systems in the power industry [15].

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