Zh.S. Kassenova
InnovativeUniversityof Eurasia, Kazakhstan
(e-mail: zhanar.kassenova@gmail.com)

Residual chemical analysis of amines used as corrosion inhibitors

Annotation

Main problem: Presence of acidic chemicals such as carbon dioxide and hydrogen sulphide, composition of production fluids triggers corrosion. Corrosion in oil and gas industry leads to deterioration of equipment since most of equipment is made of metal alloys. Application of inhibitors is one of the corrosion mitigation methods that needs to be controlled because it is important to identify optimal concentration of the chemicals in production fluids.Residual chemical analysis plays an important rolein identifying the appropriate dosage of corrosion inhibitors and its correction. It is imperative to find the most optimal concentration of amines due to the fact that both overdose and underdose could lead to equipment deterioration. The chemical analysis is hindered by complexity of mixtures that are applied in petroleum industry.

Purpose: The main purpose of this article is to find out the most effective method of residual chemical analysis for inhibitors used against sweet and sour corrosion by studying and analyzing corresponding literature review. The analysis should be carried out with robust, sensitive, and accurate instrumentation.

*Methods :*Theoretical study of composition and mechanism of amines used in oil and gas industry as corrosion inhibitors and selection of appropriate instrumental analytical techniques for the residual analysis.

Results and their importance: After careful studying and consideration of modern instrumental analytical techniques the most optimal and efficient method in terms of robustness, time saving and cost was selected. Ion chromatography is an adequate method to carry out residual chemical analysis for amines that are used as inhibitors in oil and gas industry to prevent sweet and sour corrosion.

Keywords: corrosion, oil and gas industry, residual analysis, sweet corrosion, sour corrosion, inhibitors, amines.

Introduction

One of the major problems of Oil and Gas industry is corrosion of the equipment used in this field. Schweitzer defines corrosion as deterioration of metal «by other than mechanical means» as a result of «chemical or electrochemical reaction with its environment» [1].Materials made of metal, usually carbon steel, are widely used in the industry in both downstream and upstream operations.Therefore, corrosion accompanies all activities such as drilling, production, transportation, processing, and refinery. Kermani and Harropclaim that corrosion causes 25% of all failures in petroleum production field [2]. Corrosive failure poses a serious threat to Oil and Gas production process not only in terms of financial loses but also in terms of a potential reason for environmental pollution. According to The National Physical Laboratory, 6,4% of all toxic materials are released to the environment due to corrosive failure of pipelines [3].

A common approach of corrosion scientists and engineers is to understand mechanism of the chemical reactions that lead to metal destruction. This enables to find ways of corrosion prevention and monitoring. Processes involved in Oil and Gas production are becoming more sophisticated and advanced. Meanwhile, factors contributing to corrosion issues are becoming more severe as well. Thus, corrosion is likely to remain on the highest position of troublesome difficulties of petroleum production. This article highlights corrosion caused by hydrogen sulphide and carbon dioxide that is the most common types of failure in Oil and gas industry. Typical chemicals, such as volatile amines and alkanolamines, used to prevent and slow down the process and residual chemical analysis as a method of corrosion monitoring program are discussed as well.

Apart from oxygen, the other two dissolved gases that induce corrosion are carbon dioxide (CO_2) and hydrogen sulphide (H_2S). Presence of carbon dioxide gas results in the acidification of water, thus, lowers the pH and supplies more hydrogen ions for the cathodic reaction.

$$CO_2 + H_2O \leftrightarrows H_2CO_3$$
$$H_2CO_3 \leftrightarrows H^+ + HCO_3^-$$
$$HCO_3^- \leftrightarrows H^+ + CO_3^{2-}$$
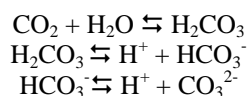
The type of corrosion attributed to the influence of carbon dioxide is called«sweet» corrosion. It is usually associated with pitting. In addition, light carboxylic acids that form as by-products usually worsen the situation by affecting the corrosion rate and making it faster. Formation water may contain some minerals that give properties of a buffer solution and prevent pH reduction.

The solubility of carbon dioxide and hence its negative effect depend on several factors, such as composition of water, temperature, and pressure. The solubility is directly proportional to pressure, and inversely proportional to temperature; i.e. carbon dioxide has higher solubility at high pressure and low temperature. Partial pressure of carbon dioxide can be used as an indicator of corrosiveness. For instance, if it exceeds 30 psi, corrosion will take place. If it is between 3 and 30 psi, corrosion is likely to occur; and the partial pressure below 3 psi is not dangerous in terms of corrosiveness.

Hydrogen sulphide does not possess corrosive properties by itself. However, hydrogen sulphide gas has high solubility in water which results in acidification and subsequent destruction of metal called «sour» corrosion. H_2S reacts with metals forming sulphides that are deposited on the surface as a black scale. In case of iron it causes additional problems because iron sulphide acts as a cathode to the iron. This results in pitting under the scale due to accumulation of favourable for corrosion conditions in a limited area. Also, «sour» corrosion is also accompanied with sulphide cracking and hydrogen induced failures like blistering and embrittlement. In the H_2S free environment, hydrogen atoms generated from the cathodic reaction collect on the metal surface. Usually it rapidly combines into hydrogen molecules that are too big to penetrate the metal and causes no harm. Presence of some substances like sulphide ions, phosphorus, arsenic compounds makes hydrogen atoms enter the metal and form hydrogen molecules inside it. This results in hydrogen induced failures of the metal.

Materials and methods

Analysis of scientific and methodological literature was taken as a basis of the study. The comparative analysis was chosen as the main method – to identify the most optimal parameters of instrumental analytical techniques for residual chemical analysis of amines in oil and gas industry.

Results

It is generally known that presence of water in the environment causes processes of corrosion. However, if water is not acidic, i.e. pH≥7, and free from hydrogen sulphide and carbon dioxide, it is considered to be non-corrosive for the petroleum production equipment. Also, in some cases hydrogen sulphide and carbon dioxideare not dangerous for the equipment if they are not dissolved in water.

So-called sweet corrosion is caused by carbon dioxide which makes water more acidic and, hence, aggressive when dissolves in it. In addition, formed carbonic acid oxidises iron and other metals.Effects of carbon dioxide on corrosive processes depend on several parameters. For example, in a condition of elevated temperaturescarbon dioxide dissolves less but high temperature can accelerate natural corrosion of metal as well. Oilfield waters have ability to keep pH at high enough level and reduce acidic effect of the dissolved gas. Formation of scales like $CaCO_3$ and $CaSO_4$ can be a sort of physical barrier for corrosive attacks if they produce solid, strong, uniform, and impermeable layers. However, if there are any breaks on the surface of the scale, corrosion will take place under the layers. According to Marcus and Mansfeld, one can distinguish corrosion caused by carbon dioxide«by the presence of smooth edged pits that are closely grouped» [4].

Hydrogen sulphide gas causes sour corrosion. Its effect on corrosive failure has the same mechanism as CO_2 but hydrogen sulphide is consideredmore aggressive. According to Schweitzer, pits that located relatively far from each other are characteristic for sour corrosion [1; 23]. Also, there is a probability of formation hydrogen as a result of cathodic reaction. It worsens the situation and may lead to further processes, such as cracking, blistering, and embrittlement. Blistering usually occurs when lower strength steels are employed because hydrogen gas pressuremakes metal structure weaker and ruptures can appear.On the other hand, high strength steel suffers from embrittlement due to its toughness which makes it more prone to cracking under physical stress. It is obvious that hydrogen induced corrosive processes are more dangerous than failures caused by H_2S itself. According to Akzo Nobel Surfactant, «hydrogen-induced failures can also be caused by sweet corrosion but it is more often associated with sour corrosion» [5].

Surprisingly, alkanolamines that widely used to remove acid compounds like hydrogen sulphide and carbon dioxide from the gas streams can be a reason themselves for corrosion of equipment. For example, Rennie claims that methanolamine (MEA) has been reported to be responsible for stress corrosion cracking failure.This type of corrosion is a result of two affecting factors: presence of corrosive environment and continuous stress either internal or external. According to Rennie,stress corrosion crackingcannot be attributed only to methanolamine by itself. The corrosion is promoted when the system is oversaturated by carbon dioxide [6]. If hydrogen sulphide ratio to carbon dioxide is large the corrosive failure is being slowed down due to suppression of active-passive transition. DuPart, Bacon, and Edwards state that stress corrosion cracking processes can be preventedby using methyl diethanolamineinstead of methanolamine for the gas treatment and apply stress relieving for welded equipments [7].

Corrosion inhibitors are chemical substances which are added to a corrodent, i.e. steam, acids, and cooling waters, in order to prevent corrosive failure of equipment and slow down reactions causing the process. Jones expresses importance of the role of chemical inhibitors by calling these compounds«the first line of defence against corrosion on the oil industry» [8]. The examples of preferentially water soluble inhibitors are quaternary amines, amine salts, and salted imidazolines. Inhibitors that are preferentially oil soluble are primary amines, phosphate esters, fatty acids, and imidazolines. Inhibitors that are soluble in oil and dispersible in water are used with special additives which help to transport the active ingredients to the wet metal surface, hence, improve their dispersibility. Inhibitor formulations vary depending on field conditions, fluid composition, and flow and the inhibitor compatibility with other chemicals and compounds must be checked. The example of

inappropriate use of an inhibitor can be fatty acids employed for fields with high content of calcium. In this case, calcium ions will form insoluble salts with fatty acids and precipitate creating more problems.

Amines belong to the organic cationic class of inhibitors widely used in petroleum production as potent agents for slowing down corrosion caused by hydrogen sulphide and carbon dioxide. They are adsorbed on the surface of metal and form protective film.There are three factors that affect film desorption into fluids contacting the metal and subsequent loss. They are time, temperature, and concentration of inhibitors in the solution. In gas production equipment the protective film usually remains longer than for oil production wells due to the fact that liquids are less transported.

One type of amine based inhibitors widely used in petroleum production are amines with short or cyclic chains or so called neutralising amines are used in oxygen free environment and especially effective for gas production due to their volatility. If the system that requires corrosion protection is closed and contains mainly steam or gas phase, volatile corrosion inhibitors (VCI) are frequently used. Morpholine and salts of low mass straight chain or cyclic structured amines are considered to be vapour phase inhibitors. The main disadvantage of VCI is a relatively thin protective film due to absence of solvated oil molecules. Therefore, the inhibitor must be supplied on a continuous basis. The fact that these inhibitors are not solvated by other oil molecules makes the protective film thinner than in case of inhibitors with long hydrocarbon chain. Therefore, volatile inhibitors should be continuously supplied with the stream. Steam volatile amines are utilised in order to protect condensate systems from carbon dioxidecorrosion which is also referred as carbonic acid corrosion in some bibliography. During this type of corrosion the metal undergoes chemical dissolution by the acid and is collected at places where condensate usually gets trapped. Neutralising amines can suppress corrosion processes caused by acid environment, i.e. carbon dioxide corrosion, due to their basic properties. According to Moiseeve and Rashevskaya, typical representatives of this type of chemicals are morpholine, cyclohexylamine, and 2-mercaptobenzothiazole.Mechanism of inhibition of neutralising amines is based on the adsorption of formed amine cations to the surface of metal. This process reduces the reactivity of the system, and their occupation of part of the surface of the metal decreases the current needed to passivate that surface [9].

Another type of amines employed in Oil and gas industry is alkanolamines, which are used in order to clean gas streams from hydrogen sulphide and carbon dioxide. These are primary, secondary, or tertiary low mass alkanolamines. According to Gonzalez-Barba and Spagnuolo, typical examples for these agents are monomethanolamine (MEA), diethanolamine (DEA), methyldiethanolamine (MDEA), triethanolamine (TEA), and monomethylethanolamine (MMEA) [10]. These chemicalsare considered to be reactant corrosion inhibitors and interact with H_2S and CO_2 through very complex exothermal reactions. Renniegives simplified forms of these reactions as follows [6; 129]:

$$H_2S + R_2NH \longleftrightarrow R_2NH_2^+ + HS^-$$
$$CO_2 + R_2NH \longleftrightarrow R_2NH_2^+ + R_2NCOO^-$$
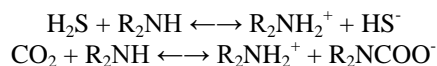
Different analytical techniques are widely used in order to collect information related to the corrosion protection. One of these methods is the analysis of residual corrosion inhibitor concentrations. According to Fortenberry, the residual methods have the advantage of speed, sensitivity, ease of setup and low cost.The corrosion rate and amount of the inhibitor in the water phase are strongly connected with each other. It is imperative to accurately measure residual concentrations in situations when the amount of aqueous phase is difficult to predict or it changes constantly.Pipelines transporting wet gas are prime examples of these kinds of systems [11].It is impossible to use corrosion monitoring methods that involves probes and coupons at low levels which are considered to be areas where the most deteriorating corrosive failure happens. Therefore, it is recommended to apply analysis of residual inhibitor concentrations for these situations. Another case where alternative methods fall to give adequate results involves multiphase pipelines that transport both oil and water. Linear polarisation techniques are useless for the given case because the system is not free from oil. On the other hand, methods ofelectrical resistance probes and weight loss coupons can be excellent alternatives to measure corrosion rate. However, these techniques take comparatively long time to be exposed to the pipeline fluids. According to Marcus and Mansfeld, claims that short exposure times for corrosion coupons cause erroneous results and suggests a minimum of 15 days [4; 56]. These factors make other methods less efficient than analysis of residual inhibitor concentrations.

Multiphase systems which contain both oil and water are frequently present in petroleum production. When applied to the system, amine inhibitors are distributedinto hydrocarbon, aqueous phases and solid phaseto the different extent.Due to the fact that corrosive failure usually happens on water wetted metal surface, one can judge about the effectiveness of the chemical inhibitorby its ability to partition into the water phase and subsequently be in a direct contact with equipment wall. The presence of solid particles in the system can cause several problems, such as creating a physical barrier for inhibitors to reach the pipe walls and consuming active parts of chemicals by adsorption mechanism with consequent loss of the inhibitors. Also, solids are considered to be extra place for bacteria to reproduce and grow. Therefore, knowledge of the partitioning of inhibitors into hydrocarbon, aqueous, and solid phases gives opportunity to determine necessary amount of the chemicals for protection. According toAchour, Blumer, and Johlman,before analysing for the inhibitor residuals in the samples, the active components of the inhibitor should be identifiedand quantified using LC/ESI-MS [12]. After

obtaining the information regarding to the individual species, each constituent components should be measured in the samples collected from the outlet of the pipeline. Observation of the inhibitor concentrationin the sample enables to determine whether the chemical is being carried along the pipe. If the active parts are absent within water phase, it indicates underdose due to total utilisation or loses through trapping. The purpose of the residual inhibitorconcentration analysis is to detect a certain trend or consistency in residual values. In addition, situations where extremely high concentrations or absolute absence of the inhibitors is detected should be carefully studied and evaluated because it is a direct indication that corrosion protection system is not valid.

There are numerous methods for corrosion inhibitor quantification that are based on separation techniques. For example, Son reports about the use of ion chromatography for ionic inhibitors like phosphonates and quats. If mass spectrometer (MS) is employed as a detector for IC, it is even possible to differentiate between individual components of ionic inhibitors [13]. It is done because usually commercial inhibitors are not pure chemicals; they can include several members of one homologous series. Gas chromatography (GC) coupled with MS is another method for simultaneous qualitative and quantitative analysis. The study of Gough, Mothershaw, and Byrne is devoted to GC/MS analysis of quats. The method incorporates addition of internal standards and sample preparation by solid phase extraction (SPE). GC/MS has benefits over other analytical methods in terms of low limit of detection (less than 1 ppm) and ability to separate and differentiate between compounds with similar structure but with different degree of branching [14].

High performance liquid chromatography (HPLC) coupled with fluorescence or UV-absorbance detector is another separation technique which was reported to be successful for corrosion inhibitor analysis. Son and Chakravarty developed method for the analysis of quaternary ammonium salts and compared the two mentioned detectors. The procedure involves isocratic flow of polar mobile phase and non-polar stationary phase. Fluorescence detector showed better performance in terms of less sensitivity for interferences. The HPLC procedure usually involves an extensive sample preparation step, such as SPE [15]. The last but not least instrumental chromatographic method for corrosion inhibitors quantification is liquid chromatography coupled with mass spectroscopy (LC/MS). Achour, Joulman and Blumer reported the use of the method for partitioning studies of the inhibitors. The instrumentation was equipped with electrospray ionisation (ESI) and operated in a positive ion mode. The analysis of the inhibitors was performed for both aqueous and oil phases. The procedure included SPE, calibration of the instrument, and characterisation of active groups of the inhibitor formulation. The LC/MS method was proven to be adequate for accurate quantification and is appropriate to be used for residual analysis [12;355]. However, it should be kept in mind, that GC/MS and LC/MS are not field instrumentations and require skilled laboratory technicians.

Discussion

As it was mentioned before, the article discusses the method of residual concentrationof amine inhibitors, such as alkanolamines and volatile amines. These amines have short aliphatic or cyclic hydrocarbon chain, and usually analysed by ion chromatography (IC). Kadnar's study was devoted to the analysis of low molecular mass alkanolamines and some volatile amines. Typical instrumentation for the analysis includes the ion chromatographic system with a conductivity detector and an integrator. Chemical micro membrane suppression system was used in Kadnar's study in order to reduce background noise and save the detector from deterioration. It is achieved by retaining ions of high conductivity. Two types of columns were employed for the analysis. An analytical column separates amines according to their affinity, and a guard column retains insoluble compounds and different impurities from the sample.Typical reagents utilised in the given experiment are tetrabutylammonium hydroxide (TBAOH) as a solution for the suppressor regeneration, hydrochloric acid as the eluent or mobile phase, standard solutions of amines for plotting calibration curve, and deionised water for the dilution. If chemical self-regeneration columns (CSRC) are used, it is advisable to utilise sulphuric acid as the eluent. Sample preparation step includes filtration of the sample in order to remove solids. Also, dilution with deionised water should be done where it is necessary to make surethatamines concentrations are within calibration scale [16].

The described system is able to separate some amines, alkali, and alkali earth metals. Ion chromatography can be appliedfor determination of some ethanolamines or short chain aliphatic amines in aqueous solution with low salinity level and steam condensates. Sometimes problems can appear when some amines have very similar retention times with alkali ions. In this case, it is advisable to change concentration of the mobile phase and try other columns for separation. Similarly, high salinity level can interfere with the analysis in the way that retention times and peak areas will be altered. The solution for this problem is careful matrix matching of standard solutions. Also, presence of alkali earth metals, such as calcium and magnesium, can deteriorate results. Due to relatively long retention times of calcium and magnium ions, broad irrelevant peaks can appear at the subsequent run and baseline drift is likely to happen. A step gradient program is useful to minimise analytical problems if amines should be determined in water samples.

Another application of IC is based on the analysis of steam volatile amine inhibitors. These amines are usually determined from condensate samples. The fact that alkaline earth metals are absent in steam condensate makes possible to have less problems during the analysis.Gilbert, Rioux, and Saheb state that hydrazineis introduced to the system together with volatile amines in order to remove dissolved oxygen and passivate metal surfaces. Mentioned compounds usually undergo chemical decomposition, especially during conditions of steam condensate systems with high temperature and pressure. The main product of the decomposition is

ammonia.Quantification of these additives and ammonia is an essential step in determining the appropriate amounts to use for maximum protection. IC has proven to be sensitive and reliable technique which shows satisfactory resolution and good linearity for determination of volatile amines and other additives.Interference from other constituents were not observed which shows highly reliable results According to their study, IC is able to provide limit of detection for morpoholine and cyclohexylamine 0,1 ppm, and for hydrazine and ammonia 0,001 ppm [17].

Conclusion

Presence ofhydrogen sulphide and carbon dioxide are two main causes of corrosion in oil and gas industry. Amines, which belong to cationic organic inhibitors, are commonly used to prevent and reduce corrosive failure of the equipment. Numerals amines and their derivatives are applied for different cases according to variable properties which depend on the structural features. Cyclic amines are utilised for gas systems due to their volatility and film forming abilities. Alkanolamines, on the other hand, are used as a H_2S and CO_2 scavengers in gas processing plants. Residual chemical analysis of inhibitors is a method of corrosion monitoring which helps to identify appropriate dosage for chemicals. Ion chromatography is an adequate, sensitive, and reliable technique for determination of concentrations of volatile amines and alkanolamines.

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Ж.С. Касенова

Инновациялық Еуразия университеті, Қазақстан

Коррозия ингибиторы ретінде қолданылатын аминдердің химиялық анализі

Көмірқышқыл газы және күкіртсутектің әсерінен өндірісте қолданылатын жабдықтардың коррозиясы өрши түседі. Мұнай және газ өндірісінде коррозия жабдықтар мен саймандардың істен

шығуына әкеледі, себебі олар көбінесе металдар балқымаларынан жасалған. Ингибиторладрды қолдану коррозияға қарсы шаралардың бірі болып табылады, бірақ оның концентрациясын міндетті түрде химиялық сараптама арқылы бақылап тұру қажет. Ингибитордың қалдық концентрациясын анықтау химиялық агенттің дұрыс дозасын есептеуде басты рөл атқарады. Аминдердің ең тиімді концетрациясын табу аса маңызды, себебі оның тым көп мөлшері, не жетіспеушілігі жабдықтың істен шығуына әкеледі. Химиялық сарапатаманы жүргізу мұнай өндіру саласындағы химиялық қоспаның күрделілігімен еркшеленеді.

Осы мақаланың басты мақсаты -қолдағы бар әдебиетті заерттеу арқылы «тәтті» және «ащы» коррозиямен күресуге көмектесетін аминдердің қалдық химиялық сараптаманың ең тиімді әдісін табу. Сараптама берік, сезімтал, дәл құралда жүргізілуі тиіс. Газды өндіру үшін жоғары ұшқыш коррозия ингибиторлары, ал мұнай өндіру үшін молекулалық массасы жоғары агенттер қолданылатындығын ескеру қажет. Сондықтан газ-кен орны үшін қысқа көмірсутекті тізбегі бар немесе циклдік құрылымы бар аминдер, ал мұнай кен орны үшін ұзын көмірсутек радикалы бар аминдер алынады.

Мұнай және газ өндіру саласында коррозия ингибиторлары ретінде қолданылатын аминдердің құрамын және механизмін теориялық түрде зерттей отырып, химиялық сарапатама үшін заманұи тиісті аспаптық талдау әдістерін салыстыра отырып, аминдердің концентрациясын анықтайытын ең тиімдісін таңдау.

Қазіргі заманғы аспапатық талдау әдістерін мұқият зерделеуден кейін, сенімділік, уақытты үнемдеу, бағасы жағынан ең тиімді әдісті таңдалды. Ионды хроматография күкірсутек пен көмірқышқыл газы тудыратын коррозияға қарсы пайдаланылатын аминдердің химиялық сарапатмасы үшін ең тиімді әдіс болып табылады. Сонымен қатар, мақалада аминдердің ионды хроматографиясын сәтті жүргізу үшін негізгі параметрлер талқыланып, ұсынылды.

Түйін сөздер: коррозия, мұнай жіне газ өндірісі, химиялық сараптама, «тәтті» коррозия, «ащы» коррозия, ингибиторлар, аминдер.

Ж.С.Касенова
Инновационный Евразийский университет, Казахстан

Остаточный химический анализ аминов, используемых в качестве ингибиторов коррозии

Присутствие компонентов, имеющих кислотный характер, таких как диоксид углерода и сероводород в производственных жидкостях могут инициировать коррозию. Коррозия является одной из главных причин выхода из строя оборудования, используемого при добыче нефти и газа. Ингибиторы коррозии применяются как один из методов борьбы с коррозией. Для того чтобы определить эффективность и рассчитать правильную дозировку химических агентов, выполняют остаточный химический анализ ингибиторов в пластовых водах. Важно подобрать оптимальную концентрацию аминов, понимая, чтои передозировка, и нехватка могут привести к поломке оборудования. Химический анализ усложняется тем, что в нефтепромысле чаще всего применяются составные химические агенты, являющиеся смесью компонентов.

Основная цельстатьи – подобрать наиболее оптимальный инструментально-аналитический метод и параметры для выполнения химического анализа аминов при лабораторном контроле ингибиторов, используемых для борьбы со «сладкой» и «кислой» коррозией. Химический анализ должен проводиться с помощью прочного, чувствительного и точного инструмента, пригодного для полевых условий. Необходимо учесть, что для добычи газа применяют легколетучие коррозионные ингибиторы, при добыче нефти - агенты с более высокой молекулярной массой. Поэтому для газового промысла берут амины с короткой углеводородной цепью или с циклической структурой, а для нефтяного промысла – амины с длинным углеводородным радикалом.

Авторами проведено теоретическое исследование состава и механизма аминов, изучена научная литература, сопоставлены современные инструментально-аналитические методы и подобраны походящие для химического анализа остаточной концентрации амины, применяемые в роли ингибиторов коррозии. После изучения химической структуры аминов, применяемых на нефтепромысле и имеющейся литературы, был сделан вывод, что наиболее эффективный метод химического анализа аминов, используемых в качестве ингибиторов против коррозии, вызываемой сероводородом и диоксидом углерода, – ионная хроматография.

Ключевые слова: коррозия, нефтегазовая отрасль, химический анализ, «сладкая» коррозия, «кислая» коррозия, ингибиторы, амины.