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PATENT ART
SCIENTISTS POWERED BY ML & DEEP WEB TOOLS

March, 2022

Technology Scouting

Sample Topic: **Monitoring Tramp Amines in Crude Oil**

www.patent-art.com

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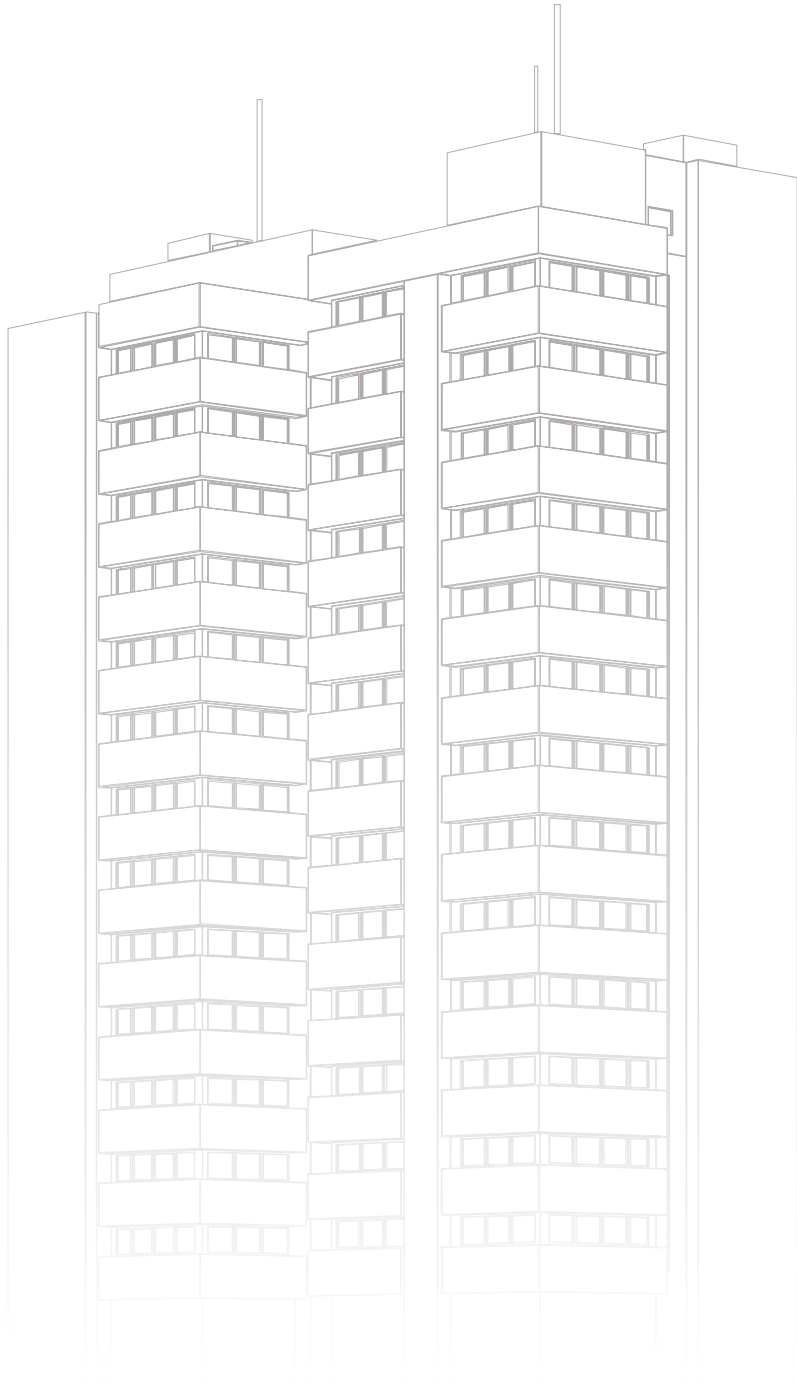

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01 About SPA family

Our family of companies

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SciTech Patent Art (SPA)

- **Technology & patent intelligence** firm established in 2002…over 20 years old
- **Hyderabad**, India–based
- **ISO 27001** certified - maintaining confidentiality is core
- **Over 300 R&D clients** in US, Japan & Europe
  - Consumer goods
  - Chemical
  - Packaging
  - Food, etc
- 100+ advanced degree **scientists and technologists. AI & Big Data analytics** team

CASESIGHT

CaseSight, Inc.

- **Consulting firm** founded in late 90s…over 25 years old
- **Boston**-based
- Focused on **patent litigation consulting**
  - Markman tutorials
  - Trial graphics
  - Expert witness preparation
  - Depositions, etc.
- Clients are highly **reputed patent litigation law firms**, e.g.,
  - Morgan Lewis
  - Mintz Levin
  - Prince Lobel
  - K&L Gates

LitAgility

LitAgility, Inc.

- **Software and related services** firm founded in 2016
- **Boston**-based
- Focused on **IP litigation strategy**
  - Contextual evidence presentation
  - Claim charts
  - SWOT analysis
  - Damages evaluation, etc.
- Used repeatedly for evaluating case strategy by litigation teams at
  - Prince Lobel
  - K&L Gates
  - Mintz Levin and others

SPA's Target – To maximize R&D's potential

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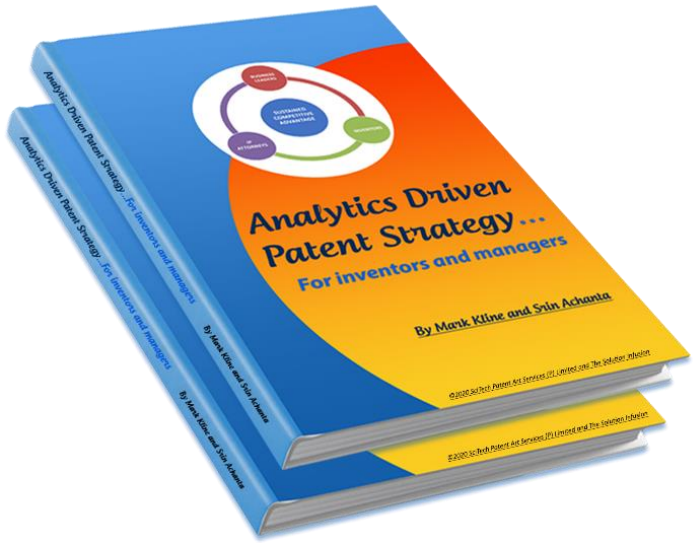
Technology Strategy & Development Process

What's been done & what's needed	How to go around art & protect my invention?	Enforce my invention & renew it
Exploration	Development	Commercialization

Robust secondary research & analysis key to success

Recommended Reading



IS YOUR **TECHNICAL TEAM** USING THE **RIGHT PATENT STRATEGY?**

...LEARN FROM THE BEST INNOVATORS...

Authors

Mark Kline

- *Retd. P&G, 30+ years in R&D*
- *125+ patents, $1B+ innovations*
- *Expert witness, IP trainer*

Srin Achanta, Ph.D.

- *P&G, Honeywell, Booz & Co.*
- *25+ years in innovation, analytics*
- *Serial entrepreneur*

Service List

Patent & Non-Patent Searches

- Prior art / landscape
- Patentability
- Freedom-to-operate
- Invalidation
- Evidence-of-use
- Design
- Alerts
- One page summaries

R&D Support Services

- Technology strategy & plan
  - Patent, non-patent & product landscape
  - Technology trends, Technology readiness, Problem-solution, tutorials, etc.
  - Whitespace analysis
- Competitor monitoring
  - Technology/product profile
  - Alerts
  - One page summaries
- Technology scouting
  - Start-up tracking
  - Partner identification
- Other custom reports

Portals

- Competitive intelligence
- Start-up / incubator tracker
- Connect-the-dots
- Non-patent literature
- Patent prosecution tracker
- Patent alerts
- Project management
- Patent portfolio analysis

Deep Web Searches

For patent invalidations, infringement detection, tracking start-ups, discovering emerging research, etc.

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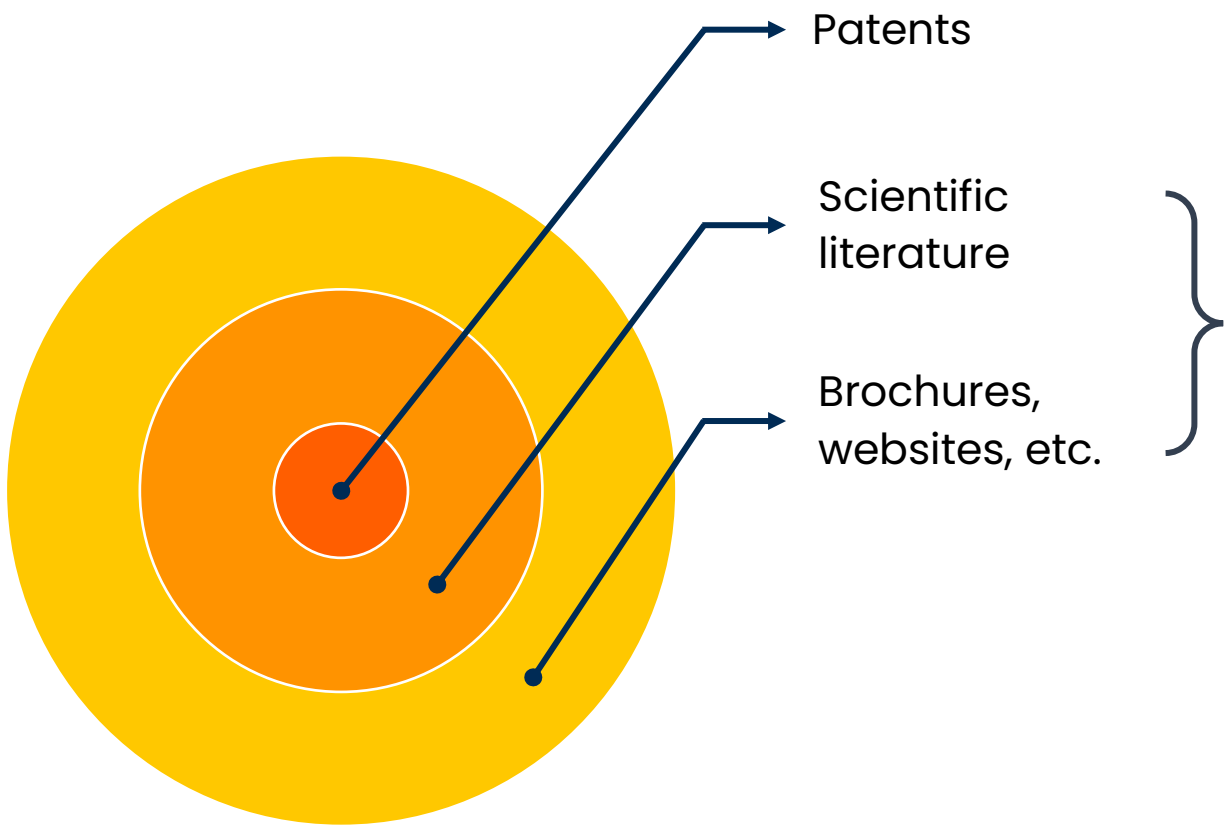
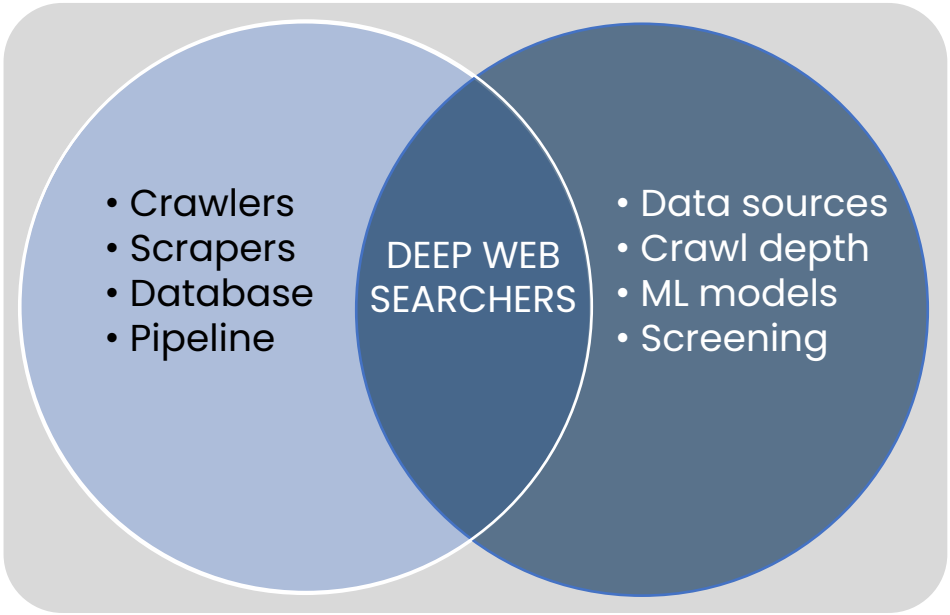
Going beyond patents to “connect-the-dots”



Deep Web Searching & Extraction



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02 Project Objective

Objective(s)

- Lower quality crude oils contain higher levels of salts (chlorides), organic chlorides, sulphur, nitrogen, metals, **tramp amines**, as well as high solids and acidity. Though cheaper, crude containing high levels of such components causes equipment corrosion
- Client is scouting for **new technologies to detect/monitor tramp amines** contained in crude oil
- Tramp amines in focus are: Monoethanolamine (MEA), Diethanolamine (DEA), Triethanolamine (TEA), 3-Methoxypropylamine (MOPA), Diglycolamine (DGA), Methyldiethanolamine (MDEA), and Monomethylamine (MMA)
- Client shared **3 industry standard methods** of interest:
  - Ion chromatography
  - Gas Chromatography coupled with Mass Spectrometry
  - Liquid Chromatography
- Client is specifically interested in learning more about the following techniques (commercialized products, state of technology development) which they assumed were **less expensive and easy to handle but also had lower accuracy**:
  - Gas Chromatography
  - Raman Spectroscopy
- Additionally, client was interested in **techniques used by specific industry leaders** who are offering technical services including online analysis of tramp amines AND potential costs of such a **service offered in Vietnam**

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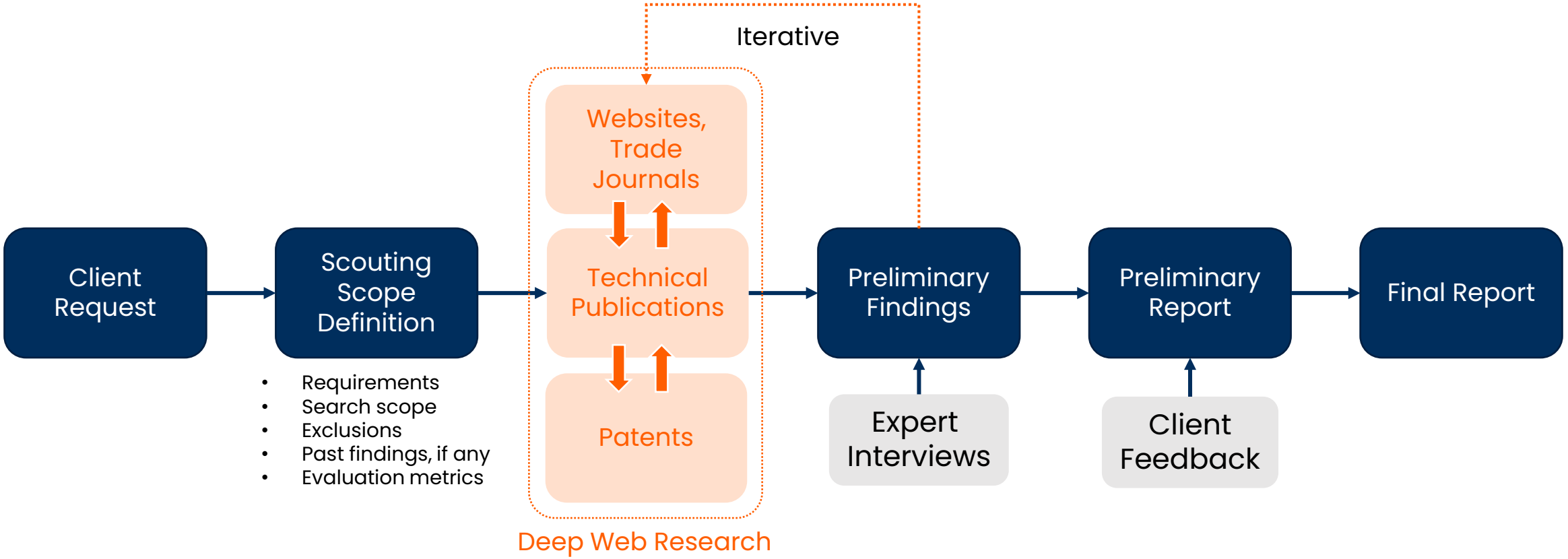
03 SPA Approach

Secondary & Primary Research Approach



Technology Evaluation Metrics

Focus tramp amines	Which of the tramp amines are detected?
Sensitivity / selectivity	What is the sensitivity and selectivity by tramp amine?
On-line / Off line	Can the method be used on-line?
Industry standard	Is the method accepted as an industry standard?
Technology Readiness Level	Is the method commercialized? If so, what's the feedback?
Cost considerations	What is the capital expense? Operating expense?
Supporting documentation	Articles, interview notes, websites, etc.

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04 Sample Findings

Summary

Analysis targets /subjects	Test methods (IC, LC, GC, Raman Spectroscopy, others)	Analysis accuracy	Laboratory / On-site (Field, Real time)	Analysis /Testing time	Features / Uniqueness	Merits
MMA	Not available (PATHFINDER Amine Test)	Improved Accuracy	Field	Significant improvement		Quick analytical turnaround time, minimizes risk to the CDU
MEA	Surface Enhanced Raman Spectroscopy/scattering (SERS)	Accurate, sensitive, and repeatable	On site or In-field	<5 Mins	Introducing a sample of industrial fluid to a device	Miniaturized analyzer, Low detection limits , CNT Ag NW substrate is approximately 10 times greater than conventional,
	Ion chromatography	Improved Accuracy	Field, Online	-NA-	Introducing a sample of industrial fluid to a device	Reduces the time of extraction, Mitigates potential surface adsorption losses
	Mass spectrometry (MS)	Improved Accuracy	Field, Online	-NA-	Introducing a sample of industrial fluid to a device	Reduces the time of extraction, Mitigates potential surface adsorption losses
	Nuclear magnetic resonance (NMR)	Improved Accuracy	Field, Online	-NA-	Introducing a sample of industrial fluid to a device	Reduces the time of extraction, Mitigates potential surface adsorption losses
	Ultra-violet (UV) spectrophotometry	Improved Accuracy	Field, Online	-NA-	Introducing a sample of industrial fluid to a device	Reduces the time of extraction, Mitigates potential surface adsorption losses
	Field asymmetric ion mobility spectrometry (FAIMS)		Onsite	1 Hour per sample	Sample preparation	Real-time screening , Portable
	Not available (PATHFINDER Amine Test)	Accurate	Field	Significant improvement		Quick analytical turnaround time, minimizes risk to the CDU
	Liquid chromatography–electrospray ionization–tandem Mass Spectrometry (LC–ESI–MS/MS)	2–10%, 1–9% and 80–120%,		-NA-	Water Sample	
	Contactless conductivity, laser induced fluorescence		Onsite	Less than about 24 hours	Introducing a sample of an industrial fluid into a device	

Interview Notes

Interview with [redacted] at Baker Hughes

- SPA contacted [redacted] Lead Engineer at Baker Hughes and had a discussion with him about Tramp amine detection.
- During the discussion [redacted] mentioned that they have tried several on-line testing methods, including that have been mentioned in the report below, [redacted] have satisfactory results.
- Hence they are trying “field asymmetric ion mobility spectrometry”. A research paper on this topic is “Fast on-site quantification and monitoring of monoethanolamine in crude oils using field asymmetric ion mobility spectrometry.
- There are a couple of patents on this topic and are part of this report
- Contact: https://www.linkedin.com/[redacted]

Interview with [redacted] at Dorf Ketal

- SPA also successfully contacted [redacted] at Dorf Ketal.
- They have experimented with many techniques and found [redacted] technique to be an ideal solution – they need to study the client situation in more detail before they can offer further advice
- They have expressed their willingness to offer such services in Vietnam
- Please contact the following person in Dorf Ketal for further details,
  - [redacted] ([redacted]@dorfketal.com)
  - He is the sales head for this region
  - https://sg.linkedin.com/[redacted]

Active Test Method Standards

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D6919-17	D7599-16 (2017)	E1151-93 (2019)
Standard Test Method for Determination of Dissolved Alkali and Alkaline Earth Cations and Ammonium in Water and Wastewater by Ion Chromatography	Standard Test Method for Determination of Diethanolamine, Triethanolamine, N-Methyldiethanolamine and N- Ethyldiethanolamine in Water by Single Reaction Monitoring Liquid Chromatography/ Tandem Mass Spectrometry	Standard Practice for Ion Chromatography Terms and Relationships
E355-96 (2021)e1	**E682-92 (2019)**	**E1642-00 (2016)**
Standard Practice for Gas Chromatography Terms and Relationships	Standard Practice for Liquid Chromatography Terms and Relationships	Standard Practice for General Techniques of Gas Chromatography Infrared (GC/IR) Analysis
D6122-21	**E1683-02 (2014)e1**	**D8321-21**
Standard Practice for Validation of the Performance of Multivariate Online, At-Line, Field and Laboratory Infrared Spectrophotometer, and Raman Spectrometer Based Analyzer Systems	Standard Practice for Testing the Performance of Scanning Raman Spectrometers	Standard Practice for Development and Validation of Multivariate Analyses for Use in Predicting Properties of Petroleum Products, Liquid Fuels, and Lubricants based on Spectroscopic Measurements

Commercialized Detectors

OndaVia Inc.

On-site monitoring ✓

Technology Platform: Raman spectroscopy

Detection chemicals: MEA and DEA

Detection Range: Mid-ppm and low-ppm

Company website: https://www.ondavia.com/

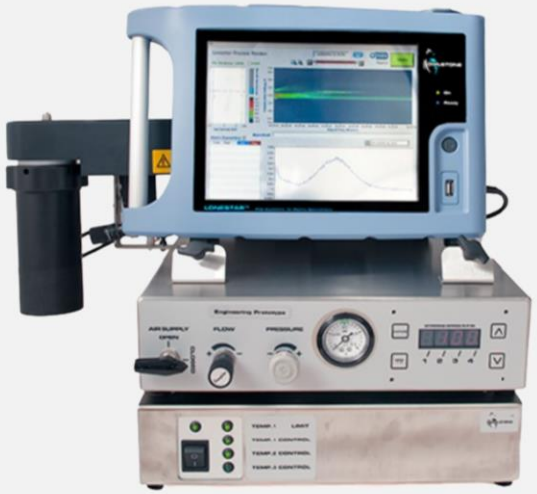
Additional informati

- The **OndaVia An** Diethanolamine DEA measuremen
- The bench-top in cartridges
- Used to control a
- The **OndaVia Ana** laboratory-grade,

Source 1, 2, 3

OWLSTONE Inc.

Continuous monitoring ✓



Technology Platform: Field asymmetric ion mobility spectrometry

Detection chemicals: Amines in both crude oil and water backgrounds

Detection Range: NA

Company website: https://www.owlstoneinc.com/technology/

Additional information:

- **Lonestar** is a **chemical monitor** in a portable, self-contained unit
- Offers the flexibility to provide both rapid alerts and **detailed sample analysis**
- Can be **trained to respond** to a broad range of **chemical scenarios**
- Can be easily **integrated with other sensors** and third-party systems to provide a complete monitoring solution
- **Suitable** for a broad variety of applications ranging from **online/at line process monitoring** to lab-based R&D

Source 1, 2

Solutions From Service Providers

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On-site Quantification of Monoethanolamine in Crude Oils

BAKER HUGHES a GE company

TOPGUARD Field Amine Measurement Services

- Field asymmetric ion mobility spectrometry (**FAIMS**) was identified as potential technology platform
- Developed customized me **determine MEA** in crude o
  - **Proprietary sample p** protocols were develo
  - **Minimized interferen** sensitivity
  - Instrument **operating optimized**
  - Proprietary data proc modeling capabilities

Features
- **On-site me**
- Portable

Manage Risks Posed By Tramp Amines

NALCO Water An Ecolab Company

PROBLEM

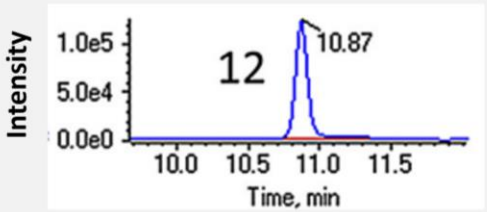
RESULTS

Source

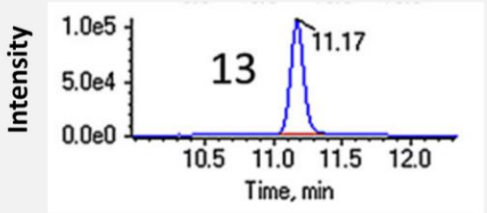
Quantification of short chain amines in aqueous matrices using LC–ESI–MS/MS

NESTE

- A new liquid chromatography–electrospray ionization–tandem Mass Spectrometry (**LC–ESI–MS/MS**) method was developed for the **determination** of more than 20 C1-C6 alkyl and **alkanolamines** in aqueous matrices
- The method employs Hydrophilic Interaction Liquid Chromatography Multiple Reaction Monitoring (**HILIC-MRM**) with a **ZIC-pHILIC column** and four stable isotope labeled amines as internal standards for signal normalization and **quantification of the amines**
- The method was validated using a **refinery process water sample** that was obtained from a cooling cycle of **crude oil distillation**
- The averaged within run precision, between run precision and **accuracy** were generally within 2–10%, 1–9% and 80–120%, respectively, depending on the analyte and concentration level
- Selected aqueous process samples were analyzed with the method



Selected ion chromatogram for methylamine detected in the non-spiked validation sample



Selected ion chromatogram for ethanolamine (62/44). detected in the non-spiked validation sample (MS/MS transition is indicated in the parenthesis if several options exist)

Source

Emerging Technologies

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1 In-situ quality monitoring of amine in natural gas sweetening units using UHF probe

Abstract: In this paper, an electrically small loop loaded with a spiral resonator is proposed for in-situ quality characterization of amine solution in gas sweetening units. The proposed UHF probe resonates at 508 MHz with a miniaturized footprint of 7.2 mm × 7.2 mm. Two different sample holder configurations, i.e. a plastic barrel tube filled with amine solution and direct immersion into the amine solution, are investigated for online quality monitoring of the amine solution. Samples of lean/ amines are collected from different units and tested under labora environment. It is found that the proposed ser system employing UHF probe exhibits st potential for the online monitoring of amine qu in the regenerative plants.

Method: Microwave-based sensing sy

Details of analysis methods: The dir real-time quality monitoring will be t direct immersion, the resonator of th applying the heat shrink over the significantly reduces the sensitivity o resonance frequency shifts to 475.79 the heat shrink. It is observed that immersed in DI-water at a depth o

Author: Zubair Akhter, Mohamed A Abou-Khousa,
Affiliation: Khalifa University of Science and Techn
Journal Details: 2019 IEEE International Instrume

2 Differentiation and quantification of corrosive amines through simple chemical process

Abstract: A new method has been developed to differentiate and quantify the amount of primary amines through a simple chemical process. Colored cyclic adduct compounds are formed by reaction of selective chemicals with primary amine. This adduct formation is preferential to the primary amine, even in the presence of a mixture of secondary and tertiary

Method: Fluorescence spectroscopy

Details of analysis methods: Colored cyclic adduct compounds are formed by reaction of selective chemicals with primary amine. This adduct formation is preferential to the primary amine, even in the presence of a mixture of secondary and tertiary amines. The adduct shows selective enhanced fluorescence emission at 475-nm wavelength under specific excitation with 420 nm. A chemical matrix, formulated with the mixture of different concentrations of primary, secondary and tertiary amines, helps to differentiate and quantify primary amines present in the mixture, even at lower concentrations

nine alone forms colored adduct complex
nine (especially ethanol amine) shows the

presence of secondary and tertiary amines
nines present in corrosive environments were tested
oned into field usable methodology

Publication Date: 29 March, 2019

2

1 Measuring concentration of analytes in liquid samples using surface-enhanced Raman spectroscopy

Abstract: A hand-held microfluidic testing device is provided that includes a housing having a cartridge receiving port and a cartridge for input to the cartridge receiving port. An optical detection system in the housing is capable of providing an illuminated electric field useful for Raman spectroscopy. The cartridge may have a sample well. The sample well is loaded with a mixture of water containing the analyte, Raman-scattering nanoparticles and a calibration solution. The calibration solution contains an analogue of the analyte differing in its Raman response, for example an isotope of the analyte. Optionally, a chemical compound capable of increasing interaction between the analyte and the nanoparticles may be added.

Method: Raman spectroscopy

Details of analysis methods: A method of measuring the amount of an analyte in water, the method comprising the steps of: obtaining a sample of water of known volume containing an analyte; mixing a known amount of an isotopologue with the sample; mixing Raman-scattering nanoparticles with the sample; mixing an amine with the sample; adjusting the pH of the mixture to 5.0 or less; producing a Raman spectrum of the mixture; and, performing a ratiometric analysis of peaks or bands within the Raman spectrum corresponding to the analyte and the isotopologue within the Raman spectrum to quantify the analyte.

Level of amine present: 20 ppb or better

Merits:
- Provide on-site or in-field analysis of aqueous samples
- Improve the accuracy of methylamine analysis
- On-site monitoring for amines, for example at a refinery, pipeline, or well-head, can help maintain appropriate corrosion protection while extending system lifetime
- Avoiding costly corrosion-induced shutdowns and failures

Demerits:
- NA

Inventors: Peterman Mark Charles, Benhabib Merwan, Ariza Carlos Atico, Kleinman Samuel Louis
Assignee: ONDAVIA
Publication #: US10895564B2
Publication Date: 2021-01-19

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05

Client Impact & Feedback

Client Impact & Feedback

"...I would like to say thanks for all. The information that you have supplied in report is extremely new and useful for us. We will have more choices of instrument which will be used to monitor amine content in the crude oil. We used to quite hesitate to choose the best method of detection amines in term of sensitivity, accuracy and cost. Before, we always think GC is the best method for qualification and quantification although it will take high cost of GC apparatus. From your information, we can take advantage of Raman spectroscopy with easy manipulation on apparatus and lower spending. Especially, the limit of detection down to ppb is our big expectation to closely monitor the pH in CDU OVHD sour water..."

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