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STATE OF NEW MEXICO
BEFORE THE WATER QUALITY CONTROL COMMISSION

IN THE MATTER OF PROPOSED NEW
RULE 20.6.8 NMAC –
Ground and Surface Water Protection –
Supplemental Requirements For Water Reuse

No. WQCC 23 - 84 (R)

NEW MEXICO ENVIRONMENT DEPARTMENT,
WATER PROTECTION DIVISION,

Petitioner.

WILDEARTH GUARDIANS' NOTICE OF INTENT TO PRESENT TECHNICAL TESTIMONY

WildEarth Guardians submits this Notice of Intent to Present Technical Testimony is submitted through its undersigned counsel. As required by NMAC 20.1.6.202 and the Hearing Officer's February 13, 2024 Pre-hearing Order ("PHO"), WildEarth Guardians provides the following:

1. Melissa Troutman will testify for WildEarth Guardians in this matter.
2. Ms. Troutman's qualifications, including a description of her education and work background are enclosed as Exhibit WG-1
3. Ms. Troutman's direct testimony is enclosed as Exhibit WG-2. Pursuant to the PHO, Ms. Troutman will provide a summary of her direct written testimony not to exceed 30 minutes during the hearing on this matter. The text of Guardians' recommended modifications to the proposed regulatory change are included in Ms. Troutman's direct written testimony.
4. WildEarth Guardians intend to offer the exhibits listed below, which are attached to this

Notice of Intent:

- **Exhibit WG-1** - Resume of Melissa Troutman
- **Exhibit WG-2** - Direct Testimony of Melissa Troutman
- **Exhibit WG-3** - WildEarth Guardians' comments submitted on December 1, 2023 - In The Matter Of Proposed New Rule 20.6.8 NMAC – Ground and Surface Water Protection – Supplemental Requirements For Water Reuse
- **Exhibit WG-4** - The New Mexico Produced Water Research Consortium, "Guidance on Produced Water Sampling Procedure"
- **Exhibit WG-5** - Pei Xu and Ryan Hall, et al., "A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water" (2021)
- **Exhibit WG-6** - Summary of Cotton Draw Unit 99 FracFocus.org disclosures
- **Exhibit WG-7** - Dusty Horwitt, J.D. and Barbara Gottlieb, Physicians for Social Responsibility, "Fracking with Forever Chemicals in New Mexico" (2023)

WildEarth Guardians requests an opportunity to provide an opening statement (10 minutes), closing statement (20 minutes), and requests to cross-examine witnesses at the hearing (time required dependent upon witnesses).

WildEarth Guardians also reserves the right to present rebuttal testimony and to introduce and move for admission of exhibits to support rebuttal testimony in this matter pursuant to the PHO.

Respectfully submitted this 15 day of April 2024,

Tim Davis
Staff Attorney
WILDEARTH GUARDIANS
301 N. Guadalupe St., Ste. 201
Santa Fe, NM 87501

Certificate of Service

I hereby certify that on April 15, 2024 a copy of the foregoing Direct Technical Testimony & Exhibits of Melissa Troutman was emailed to the persons listed below.

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<u>/s/ Tim Davis</u>
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EXHIBIT 1

MELISSA A. TROUTMAN

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PROFILE

Research & policy analyst specialized in water protection, oil/gas waste regulation, and environmental justice.

EXPERIENCE

CLIMATE & ENERGY ADVOCATE, WILDEARTH GUARDIANS – 2022 to PRESENT

Advocate for equitable climate & energy policy focused on the management of oil & gas waste, water protection, environmental justice, and community right-to-know.

COLORADO RULEMAKING CONSULTANT, YIELD – 2021 to 2022

Analyzed proposed financial assurance rule in Colorado for gaps that prevented full accountability and protection; presented findings.

RESEARCH & POLICY ANALYST, EARTHWORKS – 2018 to 2021

Authored reports & technical comments on the management of oil & gas waste operations, drafted state-level policy, tracked permits, represented before regulatory & legislative bodies, developed & coordinated multi-state coalitions.

CO-FOUNDER, PUBLIC HERALD — 2011 to 2021

Co-founded award-winning investigative news nonprofit & film production company. Led multi-year investigations, managed staff & funding, published 40+ investigative reports & open source databases.

OUTREACH COORDINATOR, MOUNTAIN WATERSHED ASSOCIATION — 2012 to 2014

Coordinated Marcellus Citizen Stewardship Project, trained community leaders, organized strategic planning summits, conducted policy analysis, testified at hearings.

STAFF REPORTER, POTTER LEADER-ENTERPRISE — 2010 to 2011

Researched, wrote, edited, photographed & designed layout of weekly regional news & natural gas development articles, coordinated with editorial and advertising staff.

PUBLIC SCHOOL TEACHER – 2006 to 2010

Taught High School English, Middle School Language Arts, & 3rd Grade in

Portsmouth, Norfolk, & Virginia Beach, VA school systems.

EDUCATION

INDIANA UNIVERSITY OF PENNSYLVANIA, 1998 - 2002 – B.A. English Literature & Theory
GPA 3.88 - Summa Cum Laude Graduate

VIRGINIA WESLEYAN COLLEGE, 2005 - 2006 – Secondary Teaching License
GPA 4.0 - Education Award, Highest GPA

CORNELL UNIVERSITY – 2021
Graduate Certificates in 1) Systems Thinking & 2) Plant Medicine

REFERENCES

Dr. Joe Stahlman, Director of Seneca Nation's Seneca-Iroquois National Museum
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AWARDS

2021 Gold Telly Award, *INVISIBLE HAND*
2021 Audience Award for *INVISIBLE HAND*, Common Good Film Festival
2020 Best Documentary Feature Award for *INVISIBLE HAND*, Hollywood Verge Film Awards
2020 Spotlight Documentary Film Gold Award for *INVISIBLE HAND*
2020 Excellence Award (Women Filmmakers), Accolade Global Film Competition
2019 Community Sentinel Award for Environmental Stewardship, FracTracker Alliance
2017 Humanitarian Award for Triple Divide [REDACTED], Accolade Global Film Competition
2016 Audience Choice Award for Triple Divide [REDACTED], Reel to Real Film Festival

2006 Education Department Award (Highest GPA), Virginia Wesleyan College

SELECTED WORKS

REPORT – Texas Oil & Gas Waste
May 2021 technical report & policy recommendations; authored for Earthworks

REPORT – California Oil & Gas Waste
January 2021 technical report & policy recommendations; authored for Earthworks

REPORT – New Mexico Oil & Gas Waste
October 2020 technical report & policy recommendations; authored for Earthworks

ARTICLE – "Dominion Energy Leaks Poison Into Pennsylvania Water Supplies"
July 2020 breaking news story with original photography; written for Public Herald

REPORT – North Dakota Oil & Gas Waste
June 2020 technical report & policy recommendations; authored for Earthworks

REPORT - Pennsylvania Oil & Gas Waste
September 2019 technical report & interactive map; authored for Earthworks

REPORT - New York Oil & Gas Waste
July 2019 technical report on impacts of oil & gas waste in NY State; authored for Earthworks

REPORT – "Still Wasting Away: The Failure to Safely Manage Oil & Gas Waste"
May 2019 technical report & policy recommendations; authored for Earthworks

REPORT SERIES – "Hidden Data Suggests Fracking Created Widespread, Systemic Impact in Pennsylvania" (January 2017) & "To Hell With Us" – Records of Misconduct Found Inside Pa. Drinking Water Investigations" (February 2017)
Seven-year investigation uncovering the systemic coverup of water contamination related to fracking development in Pennsylvania; multimedia report; written for Public Herald

ARTICLE – "Company Hasn't Released Chemical List More Than A Month After Water Contamination"
October 2015 follow up story for a series; written for Public Herald

EXHIBIT WG-2

STATE OF NEW MEXICO
BEFORE THE WATER QUALITY CONTROL COMMISSION

IN THE MATTER OF PROPOSED NEW
RULE 20.6.8 NMAC –
Ground and Surface Water Protection –
Supplemental Requirements For Water Reuse No. WQCC 23 - 84 (R)

NEW MEXICO ENVIRONMENT DEPARTMENT,
WATER PROTECTION DIVISION,

Petitioner.

DIRECT TECHNICAL TESTIMONY AND EXHIBITS

OF

MELISSA TROUTMAN

ON BEHALF OF

WILDEARTH GUARDIANS

April 15, 2024

Background and Experience

My name is Melissa Troutman and my business address is the office of WildEarth Guardians, 301 N. Guadalupe St., Suite 201, Santa Fe, NM 87501.

I am testifying on behalf of WildEarth Guardians.

I have a BA in English Literature and Education with an emphasis on literary research and analysis (2002) from Indiana University of Pennsylvania. I earned a teaching license in Secondary English and Language Arts (2006) from Virginia Wesleyan College and graduate certificates in Systems Thinking (2021) and Plant Medicine (2022) from Cornell University.

Between 2011 and 2017 I led research and reporting teams as an investigative journalist for multi-year investigations of water contamination due to oil and gas operations in Pennsylvania, guided by an Advisory Board that included nationally-renowned scientists from Duquesne University (Dr. John Stolz) and Cornell University (Dr. Anthony Ingraffea) who collectively specialize in water quality assessment, oil and gas waste field sampling and laboratory analysis, and oil and gas engineering.

In 2018, I served on Pennsylvania Representative Sara Innamorato's water protection and oil and gas waste management legislative drafting team.

I am currently a member of the New Mexico Produced Water Research Consortium and have served on several Working Groups, including Treatment Technology, Risk and Toxicology, and Public Outreach, since first joining the consortium in 2020.

A more complete description of my background, qualifications and the work in which I have been involved is included in my resume as Exhibit WG -1.

I have not appeared before the Water Quality Control Commission ("Commission" or "WQCC"). In 2020, I provided public comment to the Oil Conservation Commission pertaining to the Matter of Proposed Amendments to that Commission's Rules on Produced Water, 19.15.2, 19.15.16, and 19.15.34 New Mexico Administrative Code on behalf of Earthworks.

I have provided several other public comments on a variety of federal and state rulemakings in the past, including comments on behalf of Earthworks in association with the implementation of the Produced Water Act in New Mexico (2019 and 2020) and comments on behalf of WildEarth Guardians in association with this rulemaking (2023). My prior comments submitted for this rulemaking are included at the end of my testimony as Exhibit WG-3.

Executive Summary

I have reviewed the filing by the Water Quality Control Commission, the First Amended Petition and Statement of Reasons, and its proposed new part ("Part 8") to Title 20, Chapter 6 of the New Mexico Administrative Code ("NMAC"). Proposed by the New Mexico Environment Department (NMED) Water Protection Division (Petitioner), Part 8 is entitled "Ground and Surface Water Protection – Supplemental Requirements for Water Reuse" and will supplement the existing Ground and Surface Water Protection Regulations found at 20.6.2 NMAC.

Despite the Petitioner's revisions in March, the critical concerns detailed in WildEarth Guardians' December 1, 2023 comments on the initial petition remain unaddressed. Although the Petitioner proposes a vital and long awaited prohibition on the direct and indirect discharge of produced water, treated or untreated, to surface or groundwaters of the state, it fails to fully meet its intent "to regulate emerging methods of water reuse" in the following ways:

1. **By authorizing the utilization of produced water for demonstration and industrial projects while failing to simultaneously regulate these projects based on the best available evidence and scientific data** [see Handling, Testing, and Evaluation of Demonstration Projects below];
2. **By failing to require the full disclosure of all chemicals used in the source oil and gas wells of produced water used in demonstration and industrial projects** [see Chemical Disclosure below]; and
3. **By failing to classify the waste streams created by produced water treatment processes as non-exempt, potentially hazardous wastes** [see Hazardous Wastes below].

Adopting Part 8 without addressing these critical failures creates unnecessary exposure and pollution risks to water quality, aquatic life, the environment, workers, and the public.

Therefore, the WQCC should deny the Petitioner's proposal to adopt Part 8 until clear, adequate, and science-based standards, disclosures, and classifications are included.

Detailed Analysis

Handling, Testing, and Evaluation of Demonstration and Industrial Projects

As currently written, Part 8 authorizes the use of highly toxic oil and gas wastewater (produced water) for demonstration and industrial projects but fails to regulate these projects based on the best available evidence and scientific data that already exists, particularly within the New Mexico Produced Water Research Consortium (Consortium). By neglecting to incorporate such evidence and data, Part 8 creates new pathways for potential incidents and exposures to toxins and hazardous pollutants, including PFAS , undisclosed "trade secret" chemicals, and carcinogenic radionuclides such as Radium 226 and Radon 222, without codifying the most current scientific standards specific to these new uses in order to ensure the safest, most accurate management of produced water throughout the entire collection, transport, storage, sampling, analysis, treatment, and disposal processes.

At the very least, Part 8 must require adherence to standards set forth in "Guidance on Produced Water Sampling Procedure" by the New Mexico Produced Water Research Consortium (Consortium). This guidance is included with this testimony as Exhibit WG-4. Currently, these Consortium standards are voluntary and need to be incorporated into Part 8 to ensure a regulatory baseline for data quality and assurance.

Although these Consortium standards provide a baseline for test sampling and analysis, it must be acknowledged that they rely on EPA-approved methodologies that are unable to fully characterize the constituents of produced water. The highly toxic and variable nature of produced water and the difficulties inherent to its evaluation and treatment are thoroughly summarized in a report included in the Research Publications section of the Consortium's website[1] titled "A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water" (2021) by Consortium member Pei Xu and her team at New Mexico State University's Civil Engineering Department and Consortium member Ryan Hall at NGL Energy Water Solutions. Their report is included with this testimony as Exhibit WG–5.

Xu and Hall et al. write:

"There is a limited understanding of produced water composition due to the inherent

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[1] https://nmpwrc.nmsu.edu/research/publications.html

complexity and lack of reliable and standardized analytical methods…Despite numerous studies on [produced water] analysis, there are no standardized methods approved by the United States Environmental Protection Agency (EPA)."

The EPA has approved testing methods for specific sets of analytes – inorganic analytes (76 parameters), radioactive analytes (5 parameters), non-pesticide organic compounds (120 parameters), pesticides (70 parameters), biological indicators (8 parameters), and aquatic toxicity (4 parameters)[2] – but these tests are not specific to produced water, which as a complex mixture poses unique challenges for testing accuracy. Xu and Hall et al. acknowledge this in their report: "Since the TDS concentrations in [produced waters] may range from less than 3000 mg/L to over 300,000 mg/L, these highly saline waters are difficult to analyze. Some of the traditional analytical methods do not work accurately for an intricate water matrix like [produced water]."

Furthermore, their team's review of 157 peer-reviewed publications and regulatory standards highlighted another inadequacy of EPA-approved testing methods. They note a study by Danforth et al. which found that only 290 (24%) of 1198 unique chemical constituents identified in produced water could be quantified by EPA-approved test methods.[3]

According to reports provided on the New Mexico Environment Department (NMED) website[4] for pilot projects, project operators are using EPA-approved testing methods, which as noted above cannot detect all constituents in produced water. For example, Hall Environmental Analysis Laboratory reported that samples from the Kanalis greenhouse pilot project "were

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[2] Part Ķ, C.T. Electronic Code of Federal Regulations. 2020. Available online: https://www.ecfr.gov/cgi-bin/text-idx?node=pt40.1.136

[3] Danforth, C.; Chiu, W.A.; Rusyn, I.; Schultz, K.; Bolden, A.; Kwiatkowski, C.; Craft, E. An integrative method for identification and prioritization of constituents of concern in produced water from onshore oil and gas extraction. *Environ. Int.* **2020**, *134*, 105280.

[4] NMED Produced Water Pilot Projects, accessed 4/12/2024: https://www.env.nm.gov/new-mexico-produced-water/pilot-projects/

analyzed according to EPA procedures or equivalent."[5]

Xu and Hall et al. also write that "[d]etailed characterization of the physicochemical and biological composition of [produced water] is critical to monitor field operation and process, evaluate treatment efficiencies, assess potential risks on public health and environment during [produced water] reuse, and inform management practices and regulatory compliance." In order to do this, they recommended "[m]ulti-tiered analytical procedures" that include "field sampling; sample preservation; pretreatment techniques; basic water quality measurements; organic, inorganic, and radioactive materials analysis; and biological characterization."

According to these experts, "[a]nalytical testing is necessary throughout all stages and levels of the water cycle." Example sampling and analysis locations are illustrated in Figure 3 of their report, shown below:

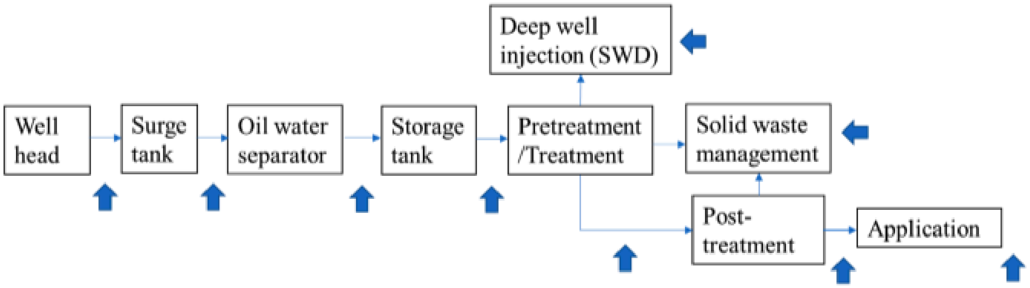

Figure 3. Example of water sampling and analysis locations and tiers.

The research team's multi-tiered approach to sampling and analysis is outlined in Table 2 of their report. It includes detailed uses, descriptions, parameters, and frequencies for each tier of testing along all stages of the treatment process, including continuous monitoring with in-line sensors (Tier 1), detailed baseline characterization and routine monitoring (Tier 2), WET,

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[5] NMED NOI, Kanalis LLC Greenhouse Pilot, accessed 4/12/2024: https://www.env.nm.gov/new-mexico-produced-water/wp-content/uploads/sites/30/2023/03/Hall-Reports-Combined-Redacted-GH-Study-Pilot.pdf

leachate and bio-mobility testing (Tier 3), and fingerprinting using biomarker and isotopic analysis (Tier 4). See the full multi-tiered approach outlined in Table 2 from their report below.

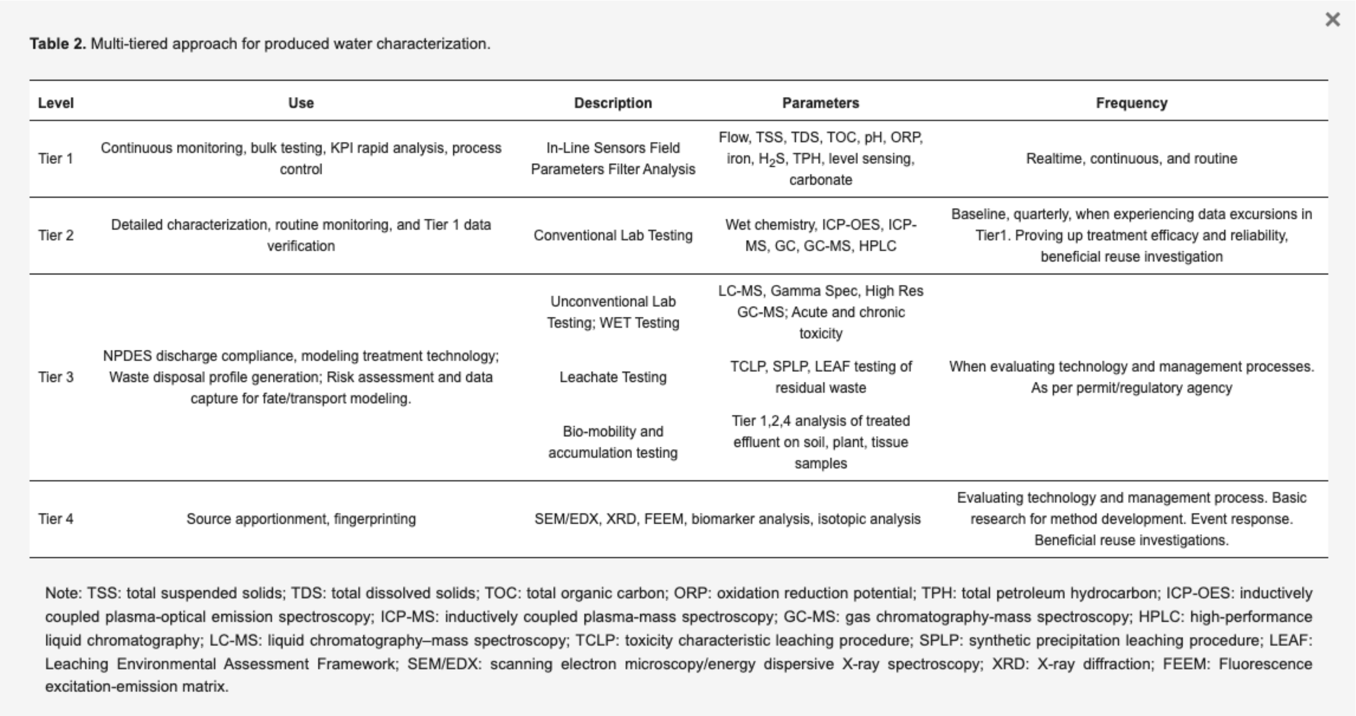

Table 2. Multi-tiered approach for produced water characterization.

Level	Use	Description	Parameters	Frequency
Tier 1	Continuous monitoring, bulk testing, KPI rapid analysis, process control	In-Line Sensors Field Parameters Filter Analysis	Flow, TSS, TDS, TOC, pH, ORP, iron, H_2S, TPH, level sensing, carbonate	Realtime, continuous, and routine
Tier 2	Detailed characterization, routine monitoring, and Tier 1 data verification	Conventional Lab Testing	Wet chemistry, ICP-OES, ICP-MS, GC, GC-MS, HPLC	Baseline, quarterly, when experiencing data excursions in Tier1. Proving up treatment efficacy and reliability, beneficial reuse investigation
Tier 3	NPDES discharge compliance, modeling treatment technology; Waste disposal profile generation; Risk assessment and data capture for fate/transport modeling.	Unconventional Lab Testing; WET Testing	LC-MS, Gamma Spec, High Res GC-MS; Acute and chronic toxicity	When evaluating technology and management processes. As per permit/regulatory agency
		Leachate Testing	TCLP, SPLP, LEAF testing of residual waste	
		Bio-mobility and accumulation testing	Tier 1,2,4 analysis of treated effluent on soil, plant, tissue samples	
Tier 4	Source apportionment, fingerprinting	SEM/EDX, XRD, FEEM, biomarker analysis, isotopic analysis		Evaluating technology and management process. Basic research for method development. Event response. Beneficial reuse investigations.

Note: TSS: total suspended solids; TDS: total dissolved solids; TOC: total organic carbon; ORP: oxidation reduction potential; TPH: total petroleum hydrocarbon; ICP-OES: inductively coupled plasma-optical emission spectroscopy; ICP-MS: inductively coupled plasma-mass spectroscopy; GC-MS: gas chromatography-mass spectroscopy; HPLC: high-performance liquid chromatography; LC-MS: liquid chromatography–mass spectroscopy; TCLP: toxicity characteristic leaching procedure; SPLP: synthetic precipitation leaching procedure; LEAF: Leaching Environmental Assessment Framework; SEM/EDX: scanning electron microscopy/energy dispersive X-ray spectroscopy; XRD: X-ray diffraction; FEEM: Fluorescence excitation-emission matrix.

In Kanalis LLC's bench project reports at NMED's pilot project website, the company notes that pre-treated produced water samples "were collected in accordance with the NMPWRC protocol, 'Guidance for Treated and Untreated Produced Water Sampling Procedure'" which incorporates some of the recommended protocols from the Xu et al. study.[6] But even though the Consortium and its members have conducted years of research to create standards and methodologies for testing and evaluation of produced water treatment technologies, these testing and evaluation standards are not yet required by state law.

In addition to the human and environmental health risks created by the failure in Part 8 to

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[6] Bench-scale Treatment Study of Produced Water from the Southern San Juan Basin New Mexico, accessed from NMED's pilot project website 4/12/2024: https://www.env.nm.gov/new-mexico-produced-water/wp-content/uploads/sites/30/2023/03/Kanalis-Final-Report-NMPWC-Approved-Vol-1.pdf

implement standards based on the latest scientific research, the Petitioner's proposal also creates a lack of quality control assurances regarding the usefulness of data generated by demonstration and industrial projects. Department staff responsible for reviewing data submitted by demonstration and industrial project operators should spend their valuable time and resources doing so knowing that the best scientific standards for handling, testing, and evaluation of produced water have already been applied as a requirement of state law.

This Part 8 rulemaking is the appropriate and necessary time to incorporate the latest science from the Consortium regarding produced water testing and evaluation, in spite of inherent deficiencies with regard to EPA-approved methodologies. Therefore, I recommend the following redline amendment to the Petitioner's criteria for demonstration and industrial project approval [20.6.8.400.B(1)(b)] under Part 8:

> (b) The Demonstration project or industrial project shall be designed to implement all sampling and evaluation protocols in "Guidance on Produced Water Sampling Procedure" published by the New Mexico Produced Water Research Consortium and provide information specific to untreated produced water quality, treatment technologies, treated produced water quality, treatment volumes, and toxicity studies for potential produced water reuse applications.

Chemical Disclosure

In addition to adding protocols based on the latest available science regarding produced water testing and evaluation procedures, the full disclosure of all chemicals used in downhole operations at the source wells for produced water is another critically needed addition to Part 8.

Without full chemical disclosure, there is even less assurance as to a) whether all chemicals have been removed by a treatment process or b) what chemicals might be present that could alter chemical composition during treatment or disrupt treatment processes. According to Linden et al. in their 2019 study for Separation Science and Technology, “Currently, Fracfocus.org contains the most comprehensive dataset on chemicals used in hydraulic fracturing; however, relatively little information is known about the additives used by production companies due to protected intellectual property and trade secrets” which “creates challenges in terms of determining the exposure safety, efficacy of treatment modalities, or the transformation products in produced water.”[7]

Simply put, if you don’t know what’s in a mixture before treatment, there is no way to know what’s there during and afterward. This critical knowledge gap threatens the validity of water quality data and protective regulations like those proposed in Part 8.

Even if all the chemicals in raw produced water are theoretically filtered out as part of the treatment process, these chemicals don’t just disappear into thin air – they end up in the waste byproducts and emissions created by produced water treatment. For example, if produced water treatment wastes are disposed of at landfills or surface waste facilities that are authorized to discharge their leachate, any chemicals that have leached from the facility can be a part of the facility’s leachate discharge as well.

The potential for produced water contaminants to travel via treatment waste to facilities with the potential to discharge leachate creates a loophole through which toxins from produced

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[7] Tiffany Liden, Inês C. Santos, Zacariah L. Hildenbrand, Kevin A. Schug, (2019) Chapter 9 - Analytical Methods for the Comprehensive Characterization of Produced Water, Separation Science and Technology, Academic Press, Volume 11, Pages 199-217. https://doi.org/10.1016/B978-0-12-815730-5.00009-0

water have the potential to threaten surface and groundwater. In order to fully protect water quality, aquatic species, and environmental and public health in New Mexico, full chemical disclosure is necessary and must be added to the stipulations for demonstration and industrial projects in Part 8. Additionally, produced water treatment wastes must be classified as non-exempt, potentially hazardous wastes to ensure they are disposed of according to their physical properties, not according to legal exemption. (Waste classification is addressed in the next section, Hazardous Wastes.)

The Devon pilot project, reports for which are available on NMED's pilot project website, exemplifies the necessity of chemical disclosure for demonstrial or industrial projects. According to NMED's NOI for Devon's pilot demonstration project, produced water will be gathered from the well pads connected by produced water gathering lines throughout the Devon Cotton Draw field, which includes Devon's Cotton Draw Unit 99 well pad. Chemical additives used for downhole operations at Cotton Draw Unit 99 are listed on FracFocus, however the specific identities of 29 of the chemicals listed are undisclosed as "confidential." The three Cotton Draw Unit 99 disclosures at FracFocus.org are summarized below and included with this testimony as Exhibit WG-6.

April 2015 – **19 "confidential" chemical ingredients** listed generically without Chemical Abstract Service (CAS) numbers, including "surfactant mixture"

May 2015 – **10 "confidential" chemical ingredients** listed generically without CAS numbers, including "surfactant mixture"

July 2015 – All chemical ingredients are disclosed with specific CAS numbers, but listed in the section not subject to 29 CFR 1910.1200(i)[8] [OSHA Hazard Communication regulation].

Of the chemicals disclosed by Devon, many can be hazardous to humans and/or aquatic life[9] including hydrochloric acid (CAS #7647-01-0), potassium hydroxide (CAS #1310-58-3), sodium persulfate (CAS #7775-27-1), 2-bromo-2-nitro-1,3-propanediol (CAS# 52-51-7), ammonium persulfate (CAS #7727-54-0), ammonium chloride (CAS# 12125-02-9), sodium hydroxide (CAS#1310-73-2), potassium metaborate (CAS# 13709-94-9), sodium perborate tetrahydrate (CAS#10486-00-7), isopropanol (CAS# 67-63-0), 1-propanesulfonic acid (CAS# 83446-68-8), sodium tetraborate (CAS# 1303-96-4), glutaraldehyde (CAS# 111-30-8), orange terpene (CAS# 68647-72-3), 2-mercaptoethanol (CAS# 60-24-2), sodium bisulfate (CAS# 7681-38-1), methanol (CAS# 67-56-1), ammonium hydroxide (CAS# 1336-21-6), propargyl alcohol (CAS# 107-19-7), 2-methyl-4-isothiazolin-3-one (CAS# 2682-20-4), acetic acid (CAS# 64-19-7), formaldehyde (CAS# 50-00-0).

Although the Consortium recommends Whole Effluent Toxicity (WET) testing for “evaluating treatment technology and management” of byproducts (see Tier 3 in Table 2 above), which is helpful in assessing overall toxicity when specific chemicals are unknown, it should be acknowledged that WET testing has several pitfalls. First, WET testing results are inherently variable and this variability cannot be eliminated.[10] Second, aquatic species subjected to WET testing in laboratories are not the same as the resident species that testing intends to protect, and

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[8] 29 CFR 1910.1200(i) outlines rules for occupational health and safety for trade secret materials, which means that 18 of the chemicals listed in this disclosure are not subject to these occupational health and safety rules. See rule here: https://www.osha.gov/laws-regs/regulations/standardnumber/1910/1910.1200

[9] These chemicals have human health hazards according to https://chemicalsafety.com/sds-search/, accessed 4/12/24.

[10] Chapman, P.M. (2000), Whole effluent toxicity testing—usefulness, level of protection, and risk assessment. Environmental Toxicology and Chemistry, 19: 3-13. https://doi.org/10.1002/etc.5620190102

the most sensitive species cannot be tested.[11] Third, controlled laboratory testing does not represent real world environments which have cumulative stressors, additional exposures, and food chain factors.[12]

Finally, WET testing relies on harming aquatic life such as fish to assess toxicity of unknown water chemistry when chemical disclosure itself could eliminate much of the uncertainty. Relying on living organisms to reveal toxicity when the disclosure of proprietary chemicals could do so more accurately raises ethical questions beyond the already "significant ethical concerns" raised by others regarding the routine use of vertebrates for WET testing.[13] This is especially true for complex chemical mixtures, like produced water, that can influence aquatic life in ways that are unmeasured by WET testing, such as "reproductive behavior, immune function, neurological function and behavior, and genetic damage."[14]

In the Devon disclosures summarized above, one of the confidential additives is listed as "surfactant mixture". Surfactants are of particular concern when assessing chemical toxicity due to the increased likelihood that they contain types of PFAS, a class of chemicals that are toxic at extraordinarily low levels, have multiple negative health effects including cancer, and are persistence in the environment – hence their nickname, "forever chemicals."

In April 2023, Physicians for Social Responsibility released "Fracking with Forever

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[11] ibid.
[12] ibid.
[13] Hughes, S.A., Maloney, E.M. and Bejarano, A.C. (2021), Are Vertebrates Still Needed in Routine Whole Effluent Toxicity Testing for Oil and Gas Discharges?. Environ Toxicol Chem, 40: 1255-1265. https://doi.org/10.1002/etc.4963
[14] Norberg-King, T.J., Embry, M.R., Belanger, S.E., Braunbeck, T., Butler, J.D., Dorn, P.B., Farr, B., Guiney, P.D., Hughes, S.A., Jeffries, M., Journel, R., Lèonard, M., McMaster, M., Oris, J.T., Ryder, K., Segner, H., Senac, T., Van Der Kraak, G., Whale, G. and Wilson, P. (2018), An International Perspective on the Tools and Concepts for Effluent Toxicity Assessments in the Context of Animal Alternatives: Reduction in Vertebrate Use. Environ Toxicol Chem, 37: 2745-2757. https://doi.org/10.1002/etc.4259

Chemicals in New Mexico" which found that "companies injected more than 8,200 [oil and gas wells] with at least one trade secret chemical per well" totalling over 240 million pounds of chemicals." Furthermore, "[o]il and gas companies injected more than 3,600 of the 8,200 wells with surfactants that could be fluorosurfactants, a class of chemical that include multiple PFAS." PSR's report is included with this testimony as Exhibit WG-7.

Furthermore, PSR's report revealed that 22 oil and gas companies currently operating in New Mexico injected a class of PFAS called PTFE, commonly known as Teflon, into 227 of their wells between 2013-2022. Devon Energy injected the most PTFE, using it in 60 wells. Devon's PTFE use accounts for more than a quarter of the 227 wells injected with PTFE by all companies, as shown in the Table 3 of PSR's report, which is included below:

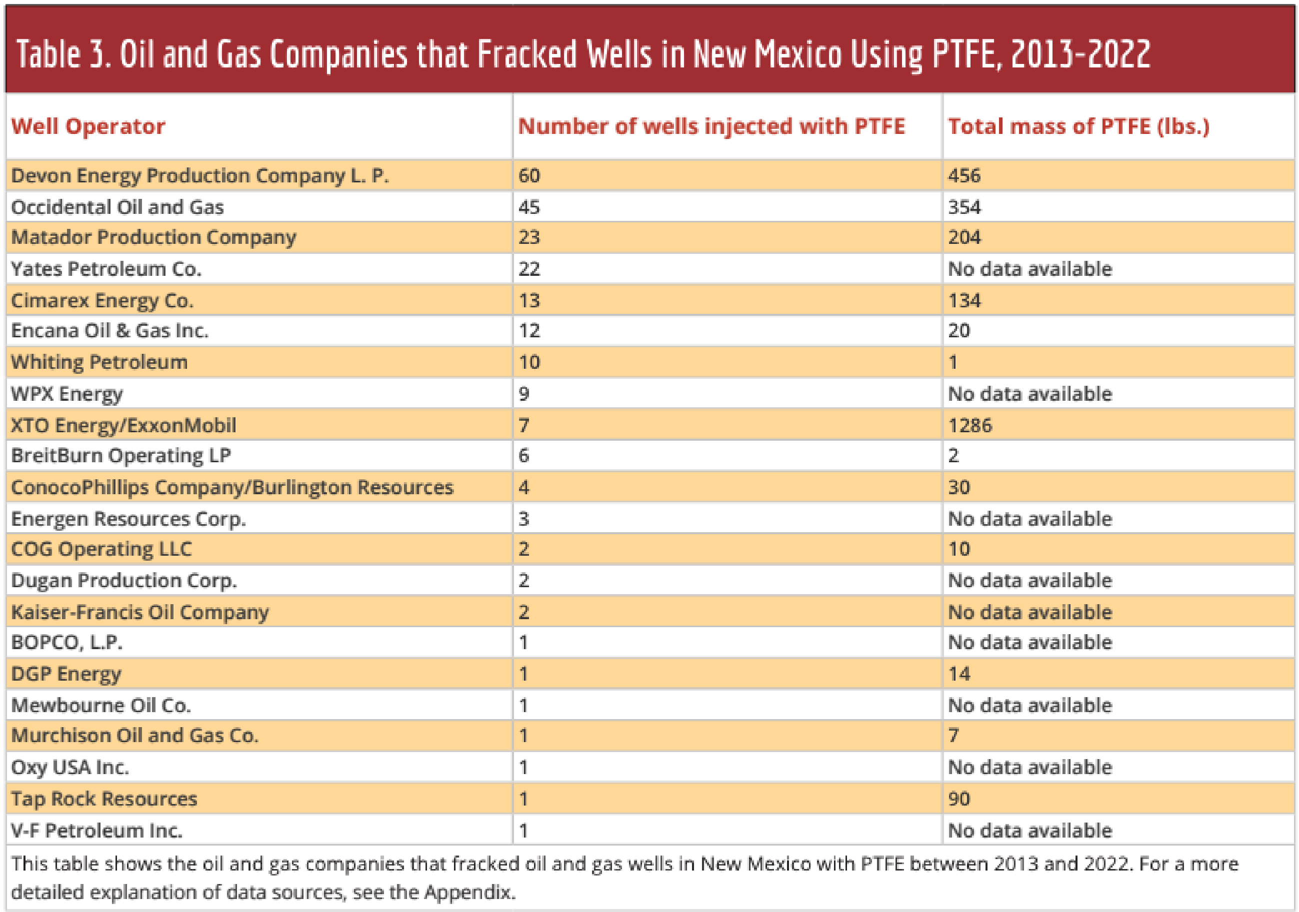

Table 3. Oil and Gas Companies that Fracked Wells in New Mexico Using PTFE, 2013-2022

Well Operator	Number of wells injected with PTFE	Total mass of PTFE (lbs.)
Devon Energy Production Company L. P.	60	456
Occidental Oil and Gas	45	354
Matador Production Company	23	204
Yates Petroleum Co.	22	No data available
Cimarex Energy Co.	13	134
Encana Oil & Gas Inc.	12	20
Whiting Petroleum	10	1
WPX Energy	9	No data available
XTO Energy/ExxonMobil	7	1286
BreitBurn Operating LP	6	2
ConocoPhillips Company/Burlington Resources	4	30
Energen Resources Corp.	3	No data available
COG Operating LLC	2	10
Dugan Production Corp.	2	No data available
Kaiser-Francis Oil Company	2	No data available
BOPCO, L.P.	1	No data available
DGP Energy	1	14
Mewbourne Oil Co.	1	No data available
Murchison Oil and Gas Co.	1	7
Oxy USA Inc.	1	No data available
Tap Rock Resources	1	90
V-F Petroleum Inc.	1	No data available

This table shows the oil and gas companies that fracked oil and gas wells in New Mexico with PTFE between 2013 and 2022. For a more detailed explanation of data sources, see the Appendix.

PTFE is just one type PFAS. According to the National Institute of Environmental Health Sciences, "PFAS are a group of nearly 15,000 synthetic chemicals, according to a chemicals database (CompTox) maintained by the U.S. Environmental Protection Agency."[15] As long as trade secret chemicals are permitted in New Mexico, particularly confidential surfactants like the ones referenced in the Devon disclosures above, there is no way to know whether PFAS is being used in oil and gas wells. This means that all waste products and spilled materials, including contaminated soils, that are generated from wells where trade secret chemicals are used must be thoroughly tested for PFAS every time to ensure these highly toxic and persistent chemicals are not present.

While trade secret chemicals are permitted in New Mexico, the state cannot confidently say it is protecting water quality, environmental health, and the public because disclosure, testing, and analysis throughout treatment and disposal will be incomplete. Part 8 must include a requirement for full chemical disclosure of all additives in any produced waters used for demonstration and industrial projects. Therefore, I recommend the following redline amendment to the Petitioner's criteria for demonstration and industrial project approval [20.6.8.400.B(1)(b)] under Part 8 in addition to the redline recommended in the previous section:

> (b) The Demonstration project or industrial project shall be designed to implement all sampling and evaluation protocols in "Guidance on Produced Water Sampling Procedure" published by the New Mexico Produced Water Research Consortium and provide information specific to untreated produced water quality,

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[15] National Institute of Environmental Health Sciences, "Perfluoroalkyl and Polyfluoroalkyl Substances (PFAS)", accessed 4/15/24 at https://www.niehs.nih.gov/health/topics/agents/pfc

including the names and Chemical Abstracts Service numbers of all ingredients in chemical additives used for operations at the source wells of produced water used by the demonstration or industrial project, treatment technologies, treated produced water quality, treatment volumes, and toxicity studies for potential produced water reuse applications. No produced water that contains withheld or confidential chemical information will be permitted for demonstration or industrial product use.

Hazardous Wastes

Produced water can be hazardous according to its physical properties. It can contain hazardous constituents such as toxic chemicals, carcinogens, radioactive material, heavy metals, ignitable and corrosive agents, and more.[16] Under the federal Resource Conservation and Recovery Act (RCRA), a substance is considered hazardous if it meets one or more of the following criteria: ignitability, corrosivity, reactivity, or toxicity.[17] However, federal hazardous waste law [40 CFR 261.4(b)(5)] explicitly exempts "[d]rilling fluids, produced waters, and other wastes associated with the exploration, development, or production of crude oil, natural gas or geothermal energy." New Mexico state law exempts the same by adoption of the federal exemption in N.M. Admin. Code 19.15.36.13(F)(1) referencing certification on form C-138.

Because produced water is classified as a non-hazardous waste by law, despite any hazardous physical properties, produced water can be handled, stored, transported, processed, cleaned up, and disposed of as if it is not a hazardous waste even if it is.

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[16] Cloelle Danforth, et al., "An integrative method for identification and prioritization of constituents of concern in produced water from onshore oil and gas extraction," Environment International, Volume 134 (2020). Available at https://doi.org/10.1016/j.envint.2019.105280.

[17] ibid.

For example, NMED's January 2023 NOI for the Tascosa Energy Partners/Hydrozonix pilot project[18] notes that it will "not discharge into or store any hazardous wastes (as defined by 40 CFR 261 and NMAC 19.15.2.7.H.3) in the [lined earthen] containment" it will use for treatment of produced water. Because produced water is exempt from hazardous waste law, this allows Tascosa to discharge [into containment] or store a potentially hazardous waste – produced water – while simultaneously saying that it won't discharge or store any hazardous waste. Here, "hazardous" is defined not by science or physical properties, but by exemption via the stroke of a pen.

The classification of produced water itself as an exempt hazardous waste is one problem in Part 8. Another problem is the classification of the wastes and byproducts, including air emissions, of the produced water treatment processes themselves.

The Petitioner's proposed disposal rules [20.6.8.400.B(h - i)] under Part 8 read:

> (h) Persons disposing of untreated or treated produced water, as part of the final disposition following a Demonstration project or industrial project, shall use one of the following methods in accordance with the relative permit: discharge to a produced water disposal well permitted pursuant to the oil conservation commission's regulations for oil and gas injection at 19.15.26 NMAC, delivery to a surface waste management facility permitted pursuant to the oil conservation commission's regulations for oil and gas surface waste management facilities (19.15.36 NMAC), or disposal in a permanent pit permitted pursuant to the oil conservation commission's regulations for oil and gas pits. closed—loop systems. below-grade tanks and sumps at 19.15.17 NMAC. The

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[18] NMED Pilot Project website, NOI for Tascosa Energy Partners/Hydronix, accessed 4/15/24 at https://www.env.nm.gov/new-mexico-produced-water/pilot-projects/

Department may consider alternative disposal options on a case-by-case basis.

(I) Persons disposing of the components of a Demonstration project or industrial project using untreated or treated produced water, as part of the final disposition must adhere to all local, state, and federal regulations, as applicable.

Despite the fact that the Petitioner's "Provisions of Part 8" declare that it "regulates the reuse of treated or untreated produced water that is ***unrelated*** to the production of oil and gas" (emphasis added), the proposed Part 8 disposal requirements reroute the wastes from demonstration and industrial "reuse" projects back to oil and gas waste disposal facilities that classify produced water and at least some treatment wastes as exempt from hazardous waste regulations. In other words, although Part 8 seems to imply that the wastes and byproducts of produced water treatment are ***not*** oil and gas wastes, they are being disposed of as such.

Exacerbating the risks of hazardous waste associated with produced water treatment is the fact that there are no emissions standards in Part 8 with regard to airborne hazards created by demonstration and industrial projects. For example, the Tascosa Energy Partners/ Hydrozonix pilot project already approved by NMED[19] treats produced water using evaporation, but there appears to be no air monitoring or sampling for hazardous air pollutants such as benzene, VOCs, or other contaminants released by the evaporative process.

Part 8 is unclear whether the wastes and byproducts from demonstration and industrial projects are classified as "exempt" or "non-exempt" from hazardous waste regulations that dictate the storage, transport and fate of these potentially hazardous materials. Therefore, I recommend the following redline amendments to the Petitioner's

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[19] ibid.

proposed disposal rules [20.6.8.400.B(h - i)] under Part 8:

(h) Persons disposing of untreated ~~or treated~~ produced water, as part of the final disposition following a Demonstration project or industrial project, shall use one of the following methods in accordance with the relative permit: discharge to a produced water disposal well permitted pursuant to the oil conservation commission's regulations for oil and gas injection at 19.15.26 NMAC, delivery to a surface waste management facility permitted pursuant to the oil conservation commission's regulations for oil and gas surface waste management facilities (19.15.36 NMAC), or disposal in a permanent pit permitted pursuant to the oil conservation commission's regulations for oil and gas pits, closed-loop systems, below-grade tanks and sumps at 19.15.17 NMAC. The Department may consider alternative disposal options on a case-by-case basis.

(i) Persons disposing of treated produced water, any treatment byproducts, or ~~of the~~ components of a Demonstration project or industrial project using untreated or treated produced water, as part of the final disposition must adhere to all local, state, and federal regulations for non-exempt, potentially hazardous waste, as applicable.

Conclusions

To protect environmental and public health, science must be in the driver's seat, especially when it comes to chemical disclosure and the management of potentially hazardous waste streams like produced water treatment wastes and byproducts. Therefore, I reaffirm the following amendments to the Petitioner's proposed rule, with justifications:

1) **The Part 8 rulemaking is the appropriate and necessary time to incorporate**

the latest science from the New Mexico Produced Water Research Consortium regarding produced water testing and evaluation, in spite of inherent deficiencies with regard to EPA-approved methodologies. Doing so will codify baseline standards based on current research for safe, accurate monitoring of field operations and processes, evaluation of treatment efficiencies, assessing potential risks on public health and environment, informing management practices and regulatory compliance, and increasing the value and validity of data generated by demonstration and industrial projects. Therefore, I recommend the following redline amendment to the Petitioner's criteria for demonstration and industrial project approval [20.6.8.400.B(1)(b)] under Part 8:

> (b) The Demonstration project or industrial project shall be designed to implement all sampling and evaluation protocols in "Guidance on Produced Water Sampling Procedure" published by the New Mexico Produced Water Research Consortium and provide information specific to untreated produced water quality, treatment technologies, treated produced water quality, treatment volumes, and toxicity studies for potential produced water reuse applications.

2) **While trade secret chemicals are permitted in New Mexico, the state cannot confidently say it is protecting water quality, environmental health, and the public because disclosure, testing, and analysis throughout treatment and disposal will be incomplete. Part 8 must include a requirement for full chemical disclosure of all additives in any produced waters used for demonstration and industrial projects.** Doing so will greatly eliminate knowledge gaps and uncertainty regarding the constituents in untreated and treated produced

water, waste products and byproducts, including PFAS. It will also inform waste management practices with regard to leachate, as both OCD permitted surface waste management facilities and NMED permitted special waste landfills accepting contaminated oil and gas waste can discharge leachate under current law. Therefore, I recommend the following redline amendment to the Petitioner's criteria for demonstration and industrial project approval [20.6.8.400.B(1)(b)] under Part 8 in addition to the redline recommended in the previous section:

> (b) The Demonstration project or industrial project shall be designed to implement all sampling and evaluation protocols in "Guidance on Produced Water Sampling Procedure" published by the New Mexico Produced Water Research Consortium and provide information specific to untreated produced water quality, including the names and Chemical Abstracts Service numbers of all ingredients in chemical additives used for operations at the source wells of produced water used by the demonstration or industrial project, treatment technologies, treated produced water quality, treatment volumes, and toxicity studies for potential produced water reuse applications. No produced water that contains withheld or confidential chemical information will be permitted for demonstration or industrial product use.

3) **Part 8 is unclear as to whether the wastes and byproducts from demonstration and industrial projects are classified as "exempt" or "non-exempt" from hazardous waste regulations that dictate the storage, transport and fate of these potentially hazardous materials. Part 8 must make this explicit.** Doing so will ensure the most safe and

appropriate waste management practices will be employed based on physical properties rather than exemption or ambiguity. Therefore, I recommend the following redline amendments to the Petitioner's proposed disposal rules [20.6.8.400.B(h - i)] under Part 8:

> (h) Persons disposing of untreated ~~or treated~~ produced water, as part of the final disposition following a Demonstration project or industrial project, shall use one of the following methods in accordance with the relative permit: discharge to a produced water disposal well permitted pursuant to the oil conservation commission's regulations for oil and gas injection at 19.15.26 NMAC, delivery to a surface waste management facility permitted pursuant to the oil conservation commission's regulations for oil and gas surface waste management facilities (19.15.36 NMAC), or disposal in a permanent pit permitted pursuant to the oil conservation commission's regulations for oil and gas pits, closed-loop systems, below-grade tanks and sumps at 19.15.17 NMAC. The Department may consider alternative disposal options on a case-by-case basis.
>
> (i) Persons disposing of treated produced water, any treatment byproducts, or ~~of the~~ components of a Demonstration project or industrial project using untreated or treated produced water, as part of the final disposition must adhere to all local, state, and federal regulations for non-exempt, potentially hazardous waste, as applicable.

I declare under penalty of perjury that the foregoing Direct Technical Testimony is true and correct based on my own personal knowledge and belief.

Dated this 15th day of April 2024.

/s/ *Melissa Troutman*
MELISSA TROUTMAN

EXHIBIT WG-3

STATE OF NEW MEXICO
ENVIRONMENTAL DEPARTMENT

WATER PROTECTION DIVISION

IN THE MATTER OF PROPOSED AMENDMENTS
TO THE DEPARTMENT'S

DRAFT RULES: *Ground and Surface Water Protection –*
Supplemental Requirements for Water Reuse (20.6.8 NMAC)

PROPOSED AMENDMENTS BY WILDEARTH GUARDIANS

These comments on New Mexico Environment Department's (NMED) draft rules for Ground and Surface Water Protection – Supplemental Requirements for Water Reuse (20.6.8 NMAC), as well as the attached proposed redline amendments, are submitted on behalf of WildEarth Guardians (Guardians).

I. Introduction

The Produced Water Act (PWA) (NMSA 1978, 70-13) and the Water Quality Act (WQA) (NMSA 1978, 74-6-4) require the Water Quality Control Commission (WQCC) to adopt regulations to be administered by NMED for protection of human health and the environment and "…for the discharge, handling, transport, storage, recycling or treatment for the disposition of treated produced water...". To this end, NMED has drafted requirements for "water reuse" that includes the potential reuse of the toxic waste byproduct of oil and gas extraction called "produced water."

Guardians suggests the following amendments to NMED's draft rule as a member of the New Mexico Produced Water Research Consortium (Consortium), serving on several committees including the Treatment and Technology Working Group, Risk and Toxicology Working Group, and Public Education and Outreach Working Group. Our Consortium work has revealed serious accountability concerns with regard to the handling, transport, storage, treatment, testing, monitoring, and potential reuse of this toxic waste outside of the oilfield related to how research has been managed within the Consortium in collaboration with NMED and the Oil Conservation Division. Our suggestions address critical gaps in oversight and management of produced water experimentation that create public and environmental health risks.

Even though NMED’s initial draft rule is predominantly prohibitive, the draft rule as written creates unnecessary risks to public and ecological health that can be eliminated by our suggested amendments. Guardians urges the department to correct these areas of deficiency in the draft rule:

1. Testing and Evaluation of Demonstration Projects
2. Transportation, Handling and Disposal of Treatment Products

II. Testing and Evaluation of Demonstration Projects

Currently, NMED’s draft rule falls woefully short of setting scientifically-based, protective criteria for demonstration projects. Neglecting to secure standards for testing, evaluation, monitoring, and reporting within the letter of the law creates a vacuum of accountability. While the Consortium has outlined some standards for testing and evaluation of produced water treatment technologies, these standards are not required by or defensible under state law. Because of the highly toxic nature of produced water and the experimental nature of pilot demonstration projects, we advise NMED to add specifications to the Notice of Intent section C(2) of the draft rule, which currently reads:

> *Persons implementing demonstration projects shall submit to the department all research results, including lab analyses of all water contaminants in the untreated produced water and treated produced water, to assist the commission in developing standards and regulations that may allow for the broader use of treated produced water in a manner that prevents water pollution and protects human health and the environment.*

If demonstration projects are to sincerely “assist the commission in developing standards,” then data submitted to the department must be scientifically sound. Data generated using faulty methodology or limited analytes is an unnecessary obstacle and waste of the department’s time. NMED could rely on the Consortium to set data standards, but this presents several pitfalls: 1) the Consortium doesn’t have legal authority to hold project managers accountable; 2) the Consortium is not funded to operate in perpetuity; 3) the Consortium has made mistakes managing pilot projects and lacks thorough oversight.

By setting its own standards for research and analysis in regulation, the department prevents spending its limited resources on reviewing messy, incomplete, or indefensible data from demonstration projects and avoids potential pitfalls from depending on Consortium oversight. This has the added benefit of saving demonstration projects themselves time and resources by providing clear standards and goals. Specific criteria for this section can be borrowed directly from Consortium standards for pilot demonstration projects, such as:

> *Pilot Demonstration Test Plan must include detailed information on…Process Operation, Sample and Data Collection, Sample and Data Analysis QA/QC, 3rd party analysis and review, process energy and mass balance, photo documentation…expected operational schedule, waste generation and management, treated water quality monitoring and analysis, assessment of public health and environment impact and risk relative to NMED and Consortium water treatment guidelines, and operational reliability of the system.*[1]

Regarding the draft rule's reference to lab analysis for water contaminants in section C(2), NMED should include a list of desired analytes based on commonly known constituents of oil and gas wastewater. This list should serve as a minimum baseline that can be expanded upon in subsequent rulemakings. To generate a list of commonly known constituents, NMED can simply borrow what's been documented by the U.S. Environmental Protection Agency (EPA) and United States Geological Survey (USGS).

As the EPA has acknowledged, produced water contains "salts, metals, radioactive materials, dissolved organic compounds, and hydraulic fracturing [fracking] chemicals and their transformation products (the result of reactions of these chemicals in the subsurface)."[2] Produced water from shale formations "typically contains high levels of TDS (salinity) and associated ionic constituents (bromide, calcium, chloride, iron, potassium, manganese, and sodium)."[3]

Studies in the New Mexico portion of the Permian Basin have shown average salinity levels of more than 89,000 mg/L in produced water, some 2.5 times higher than the 35,000

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[1] New Mexico Produced Water Research Consortium, *Guidance on Produced Water Treatment Research, Development, and Pilot-Scale Demonstration Testing and Evaluation* (Nov. 2020) available online: https://nmpwrc.nmsu.edu/_assets/public_information/Pilot-Testing-Guide_November2020_6.10.20211.pdf

[2] EPA, Hydraulic Fracturing for Oil and" Gas: Impacts from the Hydraulic Fracturing Water Cycle on Drinking Water Resources in the United States, EPA-600-R-16-236Fa at 7-1 (Dec. 2016), available at: http://www.epa.gov/hfstudy

[3] *Id.* at 7-42

mg/L characteristic of seawater.[4] With such high salinity levels, researchers have concluded that the basin has a “Low” potential for treatment, as desalination technologies required to render produced water safe for most uses is likely to be cost-prohibitive.[5]

Further, “[p]roduced water can also contain toxic materials, including barium, cadmium, chromium, lead, mercury, nitrate, selenium, and BTEX,” as well as acetone, ethylene glycol (anti-freeze), phthalates, polypropylene glycols, and dozens of other toxic chemicals.[6] Such toxics can have significant adverse impacts on human health including causing cancer[7] and disrupting the endocrine system.[8] Oil and gas companies also use per- and polyfluorinated substances (PFAS), “forever chemicals” that are very toxic at very small amounts.[9] PFAS are used in fracturing fluid mixtures that end up in produced water and other industry wastes.

Oil- and gas-bearing geologic formations also often contain naturally radioactive materials, which can be concentrated in fracking waste, such as produced water.[10] According to the EPA, radionuclides commonly found in produced water include “radium, radon, uranium, potassium and thorium,”[11] and produced water from shale formations in the Permian Basin has been shown to contain “significant levels of uranium.”[12] The extraction, storage, transportation, recycling, and reuse of produced water thus poses a serious threat of widespread radioactive contamination. As the EPA has acknowledged, “[o]nce oil and gas have been extracted from the formation, workers and members of the public may be exposed to radionuclides that are brought to the surface.”[13] As a British radiation biologist has stated, “All oil-field workers are radiation workers.”[14] They just don’t know it. “Tanks, filters, pumps, pipes, hoses, and trucks that [produced water] brine touches can all become contaminated, with the radium building up” and

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[4] Benko, K.L. & J.E. Drewes, Produced Water in the Western United States: Geographical Distribution, Occurrence, and Composition, 25 Envtl. Engineering Science No. 2, 239, 243 tbl.3 (2008)

[5] *Id.*

[6] EPA, *supra* note 2, at 7-4, 7-22 to -23.

[7] E.G. Elliott, Unconventional Oil and Gas Development and Risk of Childhood Leukemia: Assessing the Evidence, Science of the Total Environment 138 (2017)

[8] C.D. Kassotis et al., Endocrine-Disrupting Chemicals and Oil and Natural Gas Operations: Potential Environmental Contamination and Recommendations to Assess Complex Environmental Mixtures, 124 Environmental Health Perspectives 3, at 256 (Mar. 2016), available at: https://www.ncbi.nlm.nih.gov/pmc/articles/PMC4786988/pdf/ehp.1409535.pdf

[9] Physicians for Social Responsibility, “Fracking With ‘Forever Chemicals’ in New Mexico: Evidence Shows Oil and Gas Companies Have Used PFAS in New Mexico Wells; Water Risks Especially High for Groundwater-Dependent State,” available at https://psr.org/wp-content/uploads/2023/04/fracking-with-forever-chemicals-in-new-mexico.pdf

[10] EPA Radiation Waste Material from Oil and Gas Drilling, https://www.epa.gov/radtown/radioactive-waste-material-oil-and-gas-drilling (last accessed Nov. 17, 2023)

[11] *Id*.

[12] EPA, *supra* note 2, at 7-20.

[13] EPA, *supra* note 10.

[14] J. Nobel, America’s Radioactive Secret at 6, Rolling Stone (Jan. 21, 2020), available at: https://www.rollingstone.com/politics/politics-features/oil-gas-fracking-radioactive-investigation-937389/.

concentrating into a hardened and highly radioactive "scale."[15] Experts have attributed a slew of cancers among oil workers in Louisiana to on-the-job radiation exposure with 99 percent certainty.[16] Yet radioactive produced water is – even today – being piped and trucked across New Mexico, both inside and outside of the oil fields, without testing, without adequate protective equipment for exposed workers, and without an adequate regulatory scheme, free of loopholes and special exemptions, to protect against the public health and environmental threat.

Even more concerning, of more than a thousand chemicals found by scientists in produced water samples, only 14% have established toxicity values for risk assessment in the United States.[17] In other words, the toxicity of 86% of the chemicals found in produced water has never been studied.[18] Moreover, less than one-quarter of the nearly 1,200 chemicals identified in produced water can even be detected through standard analytical methods, a huge barrier to fully understanding the public health and environmental impacts of produced water reuse.[19] Because of this massive data gap, in 2019, EPA found that it lacked the data necessary to quantitatively evaluate "the potential risks associated with releases to the environment" of produced water."[20]

The Consortium cites USGS's "Common Produced Water Constituents and Concentrations" in its pilot demonstration project guidelines. These common constituents include barium, boron, bromide, HEM [n-hexane extractable material], MBAS [methylene blue active substances], radium 226 and 228, Strontium, TOC [total organic carbon], sulfate, TDS [total dissolved solids], and chloride (see Figure 3 below.)[21]

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[15] *Id.*

[16] *Id.* at 9-10

[17] C. Danforth et al., An Integrative Method for Identification and Prioritization of Constituents of Concern in Produced Water from Onshore Oil and Gas Extraction, Environment International 134, at 8 (2020)

[18] *Id.*

[19] *Id.* at 9. See also EPA, *supra* note 2, at 7-12 (explaining that studies have shown that "standard analytical methods are not adequate for detecting and quantifying the numerous organic chemicals, both naturally occurring and anthropogenic, that are now known to occur in produced water," and "advanced analytical techniques are needed to detect or quantify some analytes.")

[20] EPA, Management of Exploration, Development and Production Wastes: Factors Informing a Decision on the Need for Regulatory Action, at 5-29 (April 2019), available at: https://www.epa.gov/sites/production/files/2019-04/documents/management_of_exploration_development_and_production_wastes_4-23-19.pdf.

[21] Consortium, *supra* note 1.

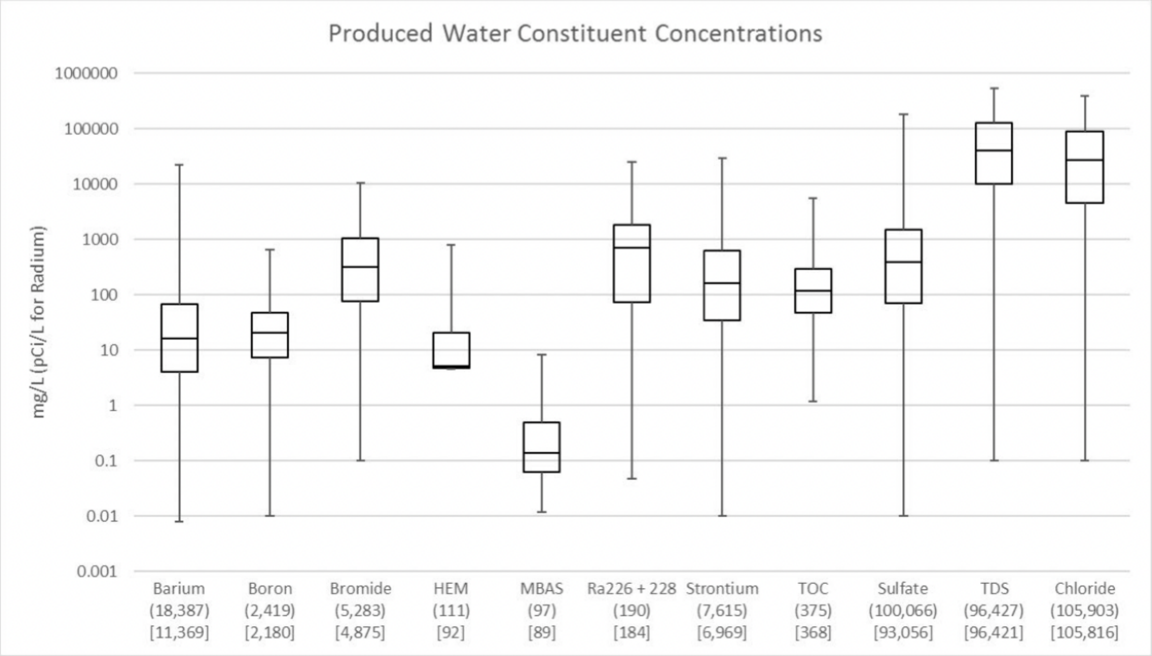

Figure 3. Example of Common Produced Water Constituents and Concentrations (USGS).

RECOMMENDATION: TESTING AND EVALUATION OF DEMONSTRATION PROJECTS

For the reasons stated above, Guardians suggests the department incorporate specificity into its Notice of Intent section C(2) and add a section C(3) that lists commonly known produced water contaminants for lab analysis. Suggested changes are shown here in red and included in our suggested redline amendments:

> *(2) Persons implementing demonstration projects shall submit to the department all research results, including lab analyses of all water contaminants in the untreated produced water and treated produced water, to assist the commission in developing standards and regulations that may allow for the broader use of treated produced water in a manner that prevents water pollution and protects human health and the environment. Submissions must include:*
>
> *(a) detailed descriptions of operation processes,*
> *(b) sample frequency and data collection methodology,*
> *(c) sample and data analysis methodology,*

(d) QA/QC for all data collection and analysis,
(e) third party analysis and review,
(f) process energy and mass balance,
(g) photo documentation,
(h) expected operational schedule,
(i) types and volumes of waste generation and management,
(j) methods of treated water quality monitoring and analysis,
(k) assessments of public health and environment impact and risk.

(3) Lab analyses of untreated and treated produced water must include but need not be limited to:

(a) all known or disclosed chemical additives, including per- and polyfluorinated substances and their precursors
(b) acknowledgement of any unknown or undisclosed, trade secret, or proprietary additives
(c) arsenic
(d) barium
(e) boron
(f) bromide,
(g) BTEX compounds
(h) chloride
(i) HEM [n-hexane extractable material],
(j) MBAS [methylene blue active substances],
(k) nitrates
(l) radium 226 and 228,
(m) strontium
(n) sulfates
(o) TOC [total organic carbon],
(p) TDS [total dissolved solids],
(q) Uranium

III. Transportation, Handling and Disposal of Treatment Products

Spills and releases of oil and gas waste, including produced water, pose serious environmental and public health risks in New Mexico. It is imperative that NMED's water reuse rules do not make an untenable situation worse. Specifically, New Mexicans deserve to know that any produced water being trucked across New Mexico for new demonstration projects is being handled safely and securely. The data shows that far too often that is not the case in the oil fields. In 2022, New Mexico's oil and gas industry reported an average of four spills of liquid contaminants *every day*, resulting in over 5.4 million gallons spilled.[22] The most common spilled material reported by industry was produced water, and the most common causes of these spills were "equipment failure" followed by "corrosion."[23]

Despite containing hazardous contaminants, oil and gas waste is generally exempt from hazardous waste law. And though it contains radioactive contaminants, oil and gas waste is not regulated like other radioactive industrial wastes, such as nuclear or medical waste. The industry enjoys other exemptions and loopholes as well, such as the Halliburton Loophole, which allows the industry to inject chemicals underground that would otherwise be regulated by the Safe Drinking Water Act.[24] Such exemptions do not, however, apply to produced water demonstration projects, as these projects are unrelated to the development or production of oil or gas. NMED should clarify that hazardous waste exemptions do not apply to the handling, transport, and disposal of produced water in demonstration projects.

The best intentions to prevent pollution do not stop produced water from spilling, whether by an oil and gas company or a produced water pilot project. In authorizing demonstration projects related to potential future use of produced water outside the oil and gas industry, NMED is creating new avenues for pollution due to unintended leaks and spills. Therefore, it's important that NMED incorporate language into the draft rule specific to the handling, transport, and disposal of produced water.

RECOMMENDATION: TRANSPORT, HANDLING & DISPOSAL OF TREATMENT PRODUCTS

NMED has the authority to require hazardous waste oversight of all byproducts from demonstration project treatment processes, including treated produced water, considering 1) oil and gas wastewater is known to contain hazardous materials, 2) pilot or demonstration projects are unrelated to the development or production of oil or gas and are therefore not exempt from

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[22] "Toxic oil and gas spills overwhelm NM regulatory agencies," WildEarth Guardians, March 2023. Available at: https://wildearthguardians.org/press-releases/toxic-oil-and-gas-spills-overwhelm-new-mexico-regulatory-agencies/

[23] *Id.*

[24] Underhill, et al., "Outcomes of the Halliburton Loophole: Chemicals regulated by the Safe Drinking Water Act in US fracking disclosures, 2014–2021," Environmental Pollution, Volume 322, 2023. Available at: https://www.sciencedirect.com/science/article/pii/S0269749122017663

hazardous waste law, and 3) treatment processes alter produced water chemistry and can introduce additional chemicals, including potentially hazardous materials.

Because of the toxic nature of produced water and the inherent risk of spills and releases from transportation and handling, all materials passed through or created by a demonstration project should be considered to be potentially hazardous until proven otherwise. Suggested language to adequately handle disposal of potentially hazardous, nonexempt materials, is offered in sections B(1)(i) and B(1)(j) of our proposed redline amendments:

> **(i) Persons disposing of treated produced water or any treatment byproduct created by a demonstration project shall provide NMED with third-party laboratory analysis of any and all potentially hazardous or radioactive contaminants prior to disposal. If lab analysis shows no hazardous or radioactive contamination, waste products may be either discharged to a produced water disposal well permitted pursuant to the oil conservation commission’s regulations for oil and gas injection (19.15.26 NMAC), delivered to a surface waste management facility permitted pursuant to the oil conservation commission’s regulations for oil and gas surface waste management facilities (19.15.36 NMAC), or disposed in a permanent pit permitted pursuant to the oil conservation commission’s regulations for oil and gas pits, closed-loop systems, below-grade tanks and sumps (19.15.17 NMAC). If lab analysis shows the presence of hazardous or radioactive contaminants, waste products will be disposed of in accordance with applicable state and federal hazardous waste regulations. Components of demonstration projects are not oil and gas operations, and are therefore not exempt from state or federal hazardous waste regulations.**
>
> **(j)** ~~(i)~~Persons disposing of the components of a demonstration project using untreated or treated produced water, as part of the final disposition must adhere to all local, state and federal regulations, as applicable**, including state and federal hazardous waste law. Components of demonstration projects are not oil and gas operations, and are therefore not exempt from state or federal hazardous waste regulations.**

The handling and transportation of all waste material from demonstration projects must be carefully documented. When lab analysis of demonstration projects wastes reveal hazardous or radioactive contamination, waste must be transported using appropriate containment with clearly displayed hazard placards on transport vehicles and storage equipment.

EXHIBIT WG-4

New Mexico Produced Water Research Consortium

GUIDANCE ON PRODUCED WATER SAMPLING PROCEDURE

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JULY, 2020

PREFACE

This guidance report was prepared by the New Mexico Produced Water Research Consortium in support of the New Mexico Environment Department and the US Environmental Protection Agency's National Water Reuse Action Plan.

The report presents a water sampling protocol for pilot demonstration of produced water treatment and use in for fit-for-purpose applications. The guidance is based on the standard methods SW-846 by Environmental Protection Agency, and methods used by commercial laboratories (e.g., Eurofins[1]) for produced water analysis.

The protocol provides step-by-step guidance and information on sample collection and preservation of produced water samples in oil and gas field. It aims to serve as a guide to the field sampling crew.

1. https://www.eurofinsus.com/media/447768/appendix-d-section-5-attachment-holdtime-container-list_2016-july.pdf

ACRONYMS

bbl	Barrels (42 gallons)
BOD_5	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
EPA	Environmental Protection Agency
HEM/SGT-HEM	n-Hexane Extractable Material/ Silica Gel Treated n-Hexane Extractable Material
HPLC	High Performance Liquid Chromatography
GC	Gas chromatography
ITRD	Innovative Technology Treatment Demonstration
NMED	New Mexico Environment Department
NMPWRC	New Mexico Produced Water Research Consortium
PPE	Personal Protection Equipment
QA/QC	Quality Assurance and Quality Control
SOP	Standard Operating Procedure
TDS	Total Dissolved Solids
TICs	Tentative Identified Compounds
TOC	Total Organic Carbon
TSS	Total Suspended Solids
TSC	Technical Steering Committee
VOA	Volatile Organic Analysis

SAMPLING PROCEDURE

Objective: provide information and guidance regarding sample collection and preservation for produced water samples in oil and gas field. Serve as a guide to the field sampling crew.

Safety: Samplers have to wear personal protection equipment, such as googles, gloves, and other personal protection equipment (PPE) required by the facility. Always work in team of two or more. Follow the safety instructions of the facility.

1. Sampling points

Sampling points and locations have to be chosen to be representative of the water samples in the facility, common sampling points include well head, surge tank, oil water separator, storage tank, points before and after treatment unit, and during applications (Figure 1). Also, the sampling points have to be consistent to assure the results from different period of measurements comparable.

Samplers need to work in teams of two or more to ensure that proper sampling techniques are followed, and adequate notes are taken at each sampling location. To prevent sample cross-contamination, samplers have to wear a new pair of disposable gloves at each sampling points and use new disposable equipment for sampling.

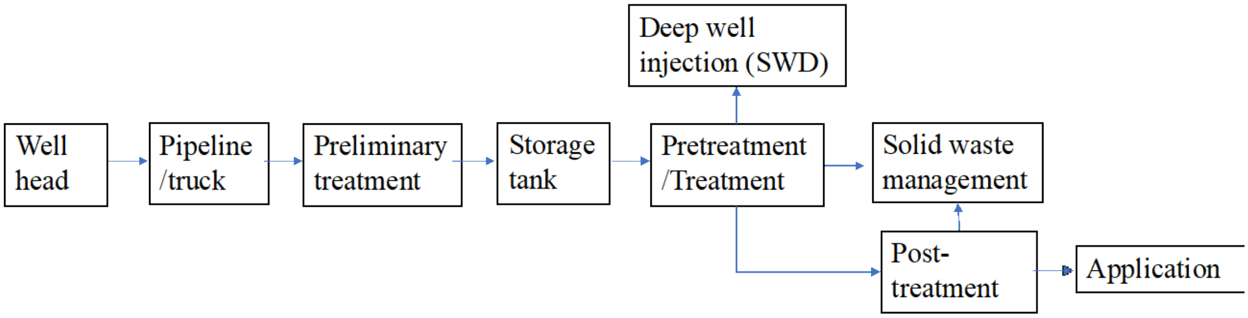

Figure 1. Common sampling points for produced water analysis.

2. Analyte selection, containers, and labels

Samplers have to carefully choose appropriate container for the target analyte. Table 1 lists the containers for different analytes. Container size can be different, but enough sample volume for analysis should be collected.
The samplers have to code each grab sample with a unique sample number and label it prior to sample collection to maintain identity and integrity. It is recommended to prepare the sample labels prior to sampling. If there is a change in sample collection procedures that are made at the site that necessitates a label change, samplers may complete labels as they collect each sample. Each self-adhesive label is completed in indelible ink and contains the following information: sampling episode, sample ID, sampling point number, sample analysis, date and time of sample collection, bottle type, analysis, and preservation.

If any of the pre-printed information is incorrect, samplers need to revise it using indelible ink. In particular, if a required preservation is not used, samplers have to mark it out and document the deviation in the sample preservation log sheet (Refer to an example in Appendix B).

Samplers will follow the following general protocols to maintain proper sample custody and to ensure that bottles do not get mixed up:

- Once the bottle label applied to the sample container, cover labels with clear tape to prevent tampering, abrasion, smearing, peeling off, or loss during transit, sample preservation or handling;
- Assemble the required sample containers for each sampling location and store them in boxes or ice chests labeled for each sampling location;
- Sample bottles have to be numbered on the lids using a permanent marker to ensure that none are missing;
- Sample bottles have to be filled in numerical order and double checked to ensure that none are skipped; and
- Samplers have to keep samples in visual sight or in locked areas at all times.

3. Sampling and preservation

3.1 Field measurement

During sampling process, several parameters should be measured onsite when each sample is collected to estimate the sample variation and to guide sample preservation methods. Samplers need to fill a 1-L glass jar during collection of each sample for field measurements.

- Temperature (SM 2550, thermometer)
- pH [pH meter or SM 4500-H B (Four color indicator strip)]
- Free residual chlorine (SM 4500-Cl G) should be measured immediately after sample collection.
- Gross density (Mass of 100 ml of sample using a scale
- Conductivity (2520 B conductivity meter)

All the meters should be calibrated before measurement based on procedures specified by the manufacturer. If a pH paper is used to measure the pH, measure the pH of the sample by transferring a drop of sample using a disposable lab-certified-clean plastic pipette onto a pH paper. The pH paper should not be inserted into the sample bottle. Free chlorine measurements will be used to guide the sample preservation.

3.2 Sampling

Samplers will collect all samples as one-time grab samples, unless other instructions are given. Depending on the sampling points, different sample collection methodology should be chosen.

Sampling from a sample tap, the first step is to sufficiently purge the sample line by opening the tap and allow water to flow into a slop bucket for a minimum of 30 seconds, or 2 to 3 gallons prior to collecting samples. After finishing this step, the water should be continuing flow into the slop bucket. Sampler will start filling sample bottles as follows:

- Retrieve a sample bottle, confirm that the bottle label already affixed to the bottle matches the location being sampled, and remove the cap. Do not touch the inside of the sample bottle or the underside of the cap.
- Introduce the sample bottle into the water stream and fill to the required level. Then replace the cap. Samplers should be careful to prevent contact of the sample tap with the inside of the sample bottle.
- Retrieve the next sample bottle and repeat the above steps until all bottles have been filled.
- Once all bottles have been filled, the contents of the slop bucket will be disposed of via the facility's drain system.

Sampling from a water tank, samplers will use a pole dipper to sample. Sample containers will be filled directly and sequentially by attaching them to the pole dipper using a zip tie. For 40-ml volatile organic analysis (VOA) vials and any other sample bottles that cannot be attached to the pole dipper, a 1-L glass transfer jar will be filled directly by attaching it to the pole dipper using zip-ties and lowering it directly into the open tank. Samplers will fill sample containers by pouring the contents of the sample transfer jar directly by tipping the pole dipper. Samplers have to minimize direct contact with the sample transfer jar and parts of the pole dipper that is lowered into the open tank.

Sampling from solid residual, sampler can collect the sample by scooping a sample directly into the sample bottles. Samplers will be careful to keep the outside of the bottle clean by using gloves and minimizing contact of the outside of the bottle and the trough contents.

For samples collected in 40-mL VOA vials, the VOA vials will be pre-preserved with HCl if needed. If free chlorine exists in a sample (Section 3.1), sodium thiosulfate will need to be added to the VOC method 624 VOA vials (Table 1). If sodium thiosulfate is needed a few crystals (10 mg/40 mL is sufficient for up to 5 part-per-million (ppm) Cl_2) will be added immediately after sample collection and prior to capping the vial.

For all VOA vials, samplers have to eliminate any headspace in the vials by first reducing water flow and collecting the sample at an angle so that the water flows gently into the vial along the inner sidewall to reduce agitation and avoid introducing air bubbles, then filling the vial to form a meniscus of water at the mouth of the vial. Cap the vial to ensure that no bubbles are present once the lid has been placed. Check to make sure that the vial does not contain bubbles by inverting the bottle several times.

After sample is collected, tighten the lid on each filled sample bottle, being careful not to over tighten. If bottle threads are dirty such that the lid is impeded from closing, clear the threads on the bottle, being careful to not introduce contamination into the sample. Clean the sample bottle with a clean, dry cloth or paper towel. Samples should be stored properly as practically possible, as discussed below. Samplers need to put sampling point description, sample information, and sample representativeness and concerns in the field sampling log sheets (refer to an example in Appendix A) at each sampling point. Sampling point pictures are also recommended if applicable.

3.3 Quality assessment samples

For quality assessment, samplers need to collect duplicate samples. The number of duplicate samples with sampling locations should be given before sampling. Samplers should collect duplicate samples as sequential grab samples. To minimize duplicate sample variability resulting from temporal variability in wastewater characteristics, the duplicate sample bottle for each analyte will be filled immediately after the original sample bottle for that analyte is filled (as opposed to filling all sample bottles for the original sample and then filling all the samples bottles for the duplicate sample). Sampling duplicate samples should follow the same procedure for original samples.

Field blanks have to be collected to evaluate potential contamination of the sample from exposure to the sampling site conditions, field handling, storage, preservation, and all analytical procedures. Field blanks will be collected in the same type of bottles for each analytical type and be analyzed for the same list of analytes. To collect field blanks, samplers will pour HPLC grade water into sampling bottles and follow the sample procedures for other samples. Equipment blanks are collected to document non-contaminated condition of sampling equipment. They are collected by rinsing sampling equipment with an analyte-free matrix (typically HPLC grade water), and in the case of tubing and similar disposable (single use) collection equipment, one equipment blank will be prepared for each lot of equipment purchased. Equipment blanks will be analyzed for the same parameters as those analyzed on the samples collected using the sampling equipment.

3.4 Preservation

Table 1 provides the sample container and preservation information for target analyte. The type and amount of preservation used have to be recorded on sample preservation log sheets (Appendix B). During sampling process, the sampling team has to confirm that the pH of samples meets the preservation requirement. If not, then the sampling team will add additional preservative to each sample to adjust the pH to meet the requirement. However, the preservation should never exceed 10 percent of the total volume.

pH adjustment for plastic bottles:

1. For samples collected in plastic bottles and that require pH preservation, samplers will measure the pH of the sample by transferring a drop of sample using a disposable lab-certified-clean plastic pipette onto a pH paper. Record value as initial pH on sample preservation log sheet

(Appendix B). If the sample does not meet the pH preservation requirement, complete step 2.

2. Add 20 drops (1 mL) of preservation chemical to every 1-liter sample using a plastic pipette dedicated to the preservation chemical. Close and tighten the sample container lid and then mix the sample. Record quantity of preservation addition on the sample preservation log sheet.
3. Repeat step 1 and 2 as needed until the target preservation is reached without exceeding 10% of the sample volume.

pH adjustment for glass bottles:

Analytical methods that require glass containers and chemical preservation include n-hexane extractable material/ Silica gel treated n-Hexane extractable materia (HEM/SGT-HEM) and samples collected in VOA vials. VOA vials will be pre-preserved with acid and then preserved for the presence of free chlorine.

HEM/SGT-HEM samples will be preserved as follows:

1. An additional 1-L glass field jar will be used to collect an extra sample that will be used to determine the amount of chemical preservation needed for a 1-L sample. This extra sample will be used only for the purpose of determining HEM/SGT-HEM preservation and then the contents will be disposed of.
2. Samplers will measure the pH of the extra sample by transferring a drop of sample using a disposable lab-certified-clean plastic pipette onto a pH paper. Record value as initial pH on sample preservation log sheet. If the sample does not meet the pH preservation requirement, continue to step 3.
3. Add 20 drops (1 mL) of preservation chemical to every 1-liter sample using a plastic pipette dedicated to the preservation chemical. Close and tighten the extra sample container lid and then mix the sample. Record quantity of preservation addition on the sample preservation log sheet.
4. Repeat step 2 and 3 as needed until the target preservation is reached without exceeding 10% of the sample volume.
5. Once the target preservation pH is met and the total volume of required preservative is known, samplers will add the same volume of acid to the HEM/SGT-HEM samples that will be sent to the lab for that sampling point. These sample jars will not be pH tested using pipettes to minimize loss of oil and grease onto the pipette.

After sampling, field sampling log sheets has to be filled to record the sampling method, sampling equipment, names of the samplers, sample collection times, field measurements, and any notes and observations.

4. Sample packing, shipping, and traffic report

If the collected samples need to be stored in cool condition (Table 1), the samples will be packed in ice chests with sufficient wet ice to maintain a temperature below 6 °C and be sent to analytical laboratories as soon as possible. Each shipment to the laboratory will contain a temperature blank, and the temperature will be taken and noted on the traffic report at the time of shipping. The temperature of the temperature blank will also be recorded by the laboratory upon receipt of samples. Exceptions include metals samples and radiological solids samples which have no temperature preservation requirements.

To maintain a record of sample collection, transfer between personnel, shipment carrier, and the laboratory, samplers will complete traffic reports for all samples sent to all laboratories. These forms are used to document sample custody transfer from the field to the laboratory.

Table 1. Analytes containers, preservations, and holding time

Analyte	**Method (Technique)**	**Sample Container**	**On-Site Preservation**	**Holding Time**
Wet Chemistry				
Total Dissolved Solids (TDS)	SM 2540 C-1997 (Gravimetric)	250 mL - Plastic	Cool to ≤ 6˚C	7 Days
Total Suspended Solids (TSS)	SM 2540 D-1997 (Gravimetric)	1000 mL - Plastic	Cool to ≤6˚C	7 Days
Specific Conductance	SM 2510 B-1997 (Conductivity Meter)	100 mL - Plastic	Cool to ≤6˚C	28 Days
Alkalinity	SM 2320 B-1997 (Titration)	250 mL - Plastic	Cool to ≤6˚C	14 Days
Chemical Oxygen Demand (COD)	EPA 410.4 (Spectrophotometric)	500 mL - Plastic	H_2SO_4 until pH < 2, Cool to ≤ 6˚C	28 Days
Total Organic Carbon (TOC)	SM 5310 B-2000 (Combustion)	250 mL – Amber Glass	H_2SO_4 or H_3PO_4 until pH < 2, Cool to ≤6˚C	28 Days
Ammonia	EPA 350.1 (Colorimetric)	250 mL - Plastic	H_2SO_4 until pH < 2, Cool to ≤ 6˚C	28 Days
N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM)	EPA 1664A (Gravimetric)	1 L - Wide-Mouth Glass	HCl or H_2SO_4 until pH < 2, Cool to ≤ 6˚C	28 Days
Biochemical Oxygen Demand (BOD5)	SM 5210 B-2001 (Titrimetric)	1000 mL - Plastic	Cool to ≤6˚C	48 Hours
Total Hardness	SM 2340 C-1997 (Titrimetric)	250 mL - Plastic	HNO3 or H_2SO_4 until pH is < 2, Cool to ≤ 6˚C	6 Months
Anions				

Fluoride, Chloride, Nitrite, Ortho-Phosphate, Bromide, Nitrate, Sulfate	ASTM D4327 (Suppressed Ion Chromatography)	500 mL - Plastic	Cool to ≤ 6˚C	28 Days except NO2, NO3, Ortho-Phosphate 48 Hours
Fluoride, Chloride, Nitrite, Ortho-Phosphate-p, Bromide, Nitrate, Sulfate Bromate, Chlorite, Chlorate	EPA 300.0 (Ion Chromatography)	500 mL - Plastic	Cool to ≤ 6˚C	28 Days except NO2, NO3, Ortho-Phosphate 48 Hours
Total Metals				
Trace elements	EPA 200.8	500 mL - Plastic	HNO_3 until pH is < 2	6 Months
Mercury	EPA 245.1 or 245.2 (Cold Vapor Atomic Absorption)	500 mL - Plastic	HNO_3 until pH is < 2	28 Days
Hexavalent Chromium	SM 3500-Cr B-2009 (Colorimetric)	250 mL - Plastic	Cool to ≤ 6˚C	24 Hours
Organics				
Diesel Range	EPA 3520C (sample preparation) EPA 8015C (analysis) (GC)	1-L - Amber Glass	Cool to ≤ 6˚C	7 Days
Gasoline Range	EPA 5030B (sample preparation) EPA 8015C (analysis) (GC)	40-mL VOA vials	Cool to ≤ 6˚C	7 Days
Volatile Organic Compounds + Tentative Identified compounds (TICs)	EPA 5030 or EPA 5035/8260C (GC /MS)	40-mL VOA vials	HCl until pH < 2, Cool to ≤ 6˚C	14 Days
Volatile Organic Compounds + TICs	EPA 624 (GC /MS)	40-mL VOA vials	HCl until pH < 2, Cool to ≤ 6˚C, Add $Na_2S_2O_3$ (a few crystals) in the presence of residual chlorine	14 Days

Semivolatile Organic Compounds + TICs	EPA 3520C/8270D (GC /MS)	1-L - Amber Glass	Cool to ≤ 6˚C	7 Days
Semivolatile Organic Compounds + TICs	EPA 625 (GC)	1-L - Glass	Cool to ≤ 6˚C, Add $Na_2S_2O_3$in the presence of residual chlorine	7 Days
Alcohols	EPA 8260C, 8270D, and 8015C (GC/MS)	40-mL VOA vials	HCl until pH < 2, Cool to ≤ 6˚C	14 Days
Oil & Grease	EPA 1664B (Extraction and Gravimetry)	1-L Amber Glass	HCl or H_2SO_4 until pH < 2, Cool to ≤ 6˚C	28 Days
Total Petroleum Hydrocarbons	EPA 1664B (Extraction and Gravimetry)	1-L Amber Glass	HCl or H_2SO_4 until pH < 2, Cool to ≤ 6˚C	28 Days
Total Petroleum Hydrocarbons by GC	Modified EPA 8100 (GC)	1-L Amber Glass	HCl until pH < 2, Cool to ≤ 6˚C	14 Days
Radioactive				
Total Radium 226 (Liquid Samples)	EPA 903.1 (Radon Emanation)	1-L - Plastic	HNO_3 until pH is < 2	6 Months
Total Radium 228 (Liquid Samples)	EPA 904.0 (Radiochemical/Precipitation)	1-L - Plastic	HNO_3 until pH is < 2	6 Months
Total Radium 226 and 228 (Solid Samples)	EPA 901.1 (Gamma Spectroscopy)	215 grams - Wide-Mouth Plastic	None	6 Months
Gross Alpha/Beta (Liquid Samples)	EPA 900.0 (Evaporation)	500 mL – Wide-Mouth Plastic	HNO_3 until pH is < 2	6 Months
Gross Alpha/Beta (Solid Samples)	EPA 900.0 (Evaporation)	30 grams - Wide-Mouth Plastic	None	6 Months
Whole Effluent Toxicity (WET)				

Acute Nonvertebrate	*Ceriodaphnia dubia* EPA 2002.0	4-L - Plastic Cubitainer	Cool to ≤ 6˚C	36 Hours
Acute Vertebrate	*Pimephales promelas* EPA 2000.0	4-L - Plastic Cubitainer	Cool to ≤ 6˚C	36 Hours
Chronic Nonvertebrate	*Ceriodaphnia dubia* EPA 1002.0	4-L - Plastic Cubitainer	Cool to ≤ 6˚C	36 Hours
Chronic Vertebrate	*Pimephales promelas* EPA 1000.0	4-L - Plastic Cubitainer	Cool to ≤ 6˚C	36 Hours

Appendix A. Field Sampling Log Sheet

Samplers’
Names: ____

Sampling
Episode: ____

Sampling Method and Sampling Equipment Used:

____

____

____

Sample ID	Date and Time	Temp (ºC)	pH	Conductivity	Weight of 100 mL of Sample	Free Chlorine

Notes: (include observations of odor and color of each aliquot, take photographs, and note any deviations from the plan):

____

____
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Appendix B. Sample Preservation Log Sheet

<table>
<tr><td colspan="8">Preservation Chemicals - List Strength of Solution from Bottle</td></tr>
<tr><td colspan="8">HCl ______ HNO_3 _________ H_2SO_4 _________ $Na_2S_2O_3$ __________NaOH __________
Other_________</td></tr>
<tr><td>Sample Number</td><td>Analysis</td><td>Date</td><td>Name of Sampler</td><td>Chemical</td><td>Initial pH</td><td>Final pH</td><td>Number of Drops</td></tr>
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EXHIBIT WG-5

water

MDPI

Review

A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water

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Abstract: Produced water is the largest waste stream associated with oil and gas production. It has a complex matrix composed of native constituents from geologic formation, chemical additives from fracturing fluids, and ubiquitous bacteria. Characterization of produced water is critical to monitor field operation, control processes, evaluate appropriate management practices and treatment effectiveness, and assess potential risks to public health and environment during the use of treated water. There is a limited understanding of produced water composition due to the inherent complexity and lack of reliable and standardized analytical methods. A comprehensive description of current analytical techniques for produced water characterization, including both standard and research methods, is discussed in this review. Multi-tiered analytical procedures are proposed, including field sampling; sample preservation; pretreatment techniques; basic water quality measurements; organic, inorganic, and radioactive materials analysis; and biological characterization. The challenges, knowledge gaps, and research needs for developing advanced analytical methods for produced water characterization, including target and nontarget analyses of unknown chemicals, are discussed.

Keywords: produced water; water quality; hydraulic fracturing; analytical methods; treatment; reuse

check for updates

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1. Introduction

In 2020, the United States became a net energy exporter and will remain so until 2050, according to the U.S. Energy Information Administration [1]. Water resource management significantly influences the oil and gas (O&G) industry because water is used for almost all stages in fossil fuel production, such as well drilling and completion, reservoir management, enhanced oil recovery, and hydraulic fracturing (HF) [2]. HF uses a large volume of water to extract O&G from an "unconventional play" (or "tight oil play"), which refers to the low permeable unconventional shale that cannot be explored and produced by conventional processes relying on the natural pressure of the wells and pumping operation [3,4].

At the initial stage of HF, fracturing fluids are injected into deep wells under high pressure to fracture the geological formation, increase permeability, and extract oil and gas. Around 91–94% (mass percentage) of the fracturing fluid is water, with ~5–8% proppant (mostly sand) and ~1% chemical additives [5,6]. After HF, a portion of injected water returns to the surface with high levels of dissolved solids, salts, and chemical additives; this water is often referred to as flowback water (FW). FW usually occurs in the first several weeks and before the well is placed in production. Over time, FW diminishes and is replaced with formation water native to the well, which is referred to as produced water (PW), occurring throughout the life of a well [7]. In the field, FW and PW are commonly co-mingled so that these streams cannot practically be distinguished. Thus, PW is often broadly defined to include both water streams.

The United States produces an estimated 900 billion gallons of produced water (PW) annually, making it the largest waste stream associated with O&G activity [8]. The amount of PW generated per year keeps increasing with unconventional O&G development (UD), which produced more than 50% of crude oil and natural gas in 2019 [1]. The production increase of the UD in the U.S. is mainly from seven key oil and gas basins: Appalachia including Marcellus and Utica (Pennsylvania, Ohio, and West Virginia), Bakken (North Dakota and Montana), Eagle Ford (South Texas), Haynesville (Louisiana and East Texas), Niobrara (Colorado and Wyoming), and the Permian Basin (West Texas and Southeast New Mexico) [9]. The PW from unconventional reservoirs usually cannot be reinjected into the shale reservoir for enhanced recovery but needs appropriate disposal such as into non-oil-producing geologic intervals (e.g., salt water disposal (SWD) wells) [3] or reuse for HF [10]. Approximately 55% of PW generated from conventional and unconventional activities in the U.S. is handled as wastewater for disposal [10]. With the increase in UD, disposal of PW in SWD wells has not only raised concern about surface water and groundwater contamination [11–13] but also risks of increased seismic events [14], such as the UD-related earthquake in the Bend Arch-Fort Worth Basin in northern Texas in 2008 [15]. In addition, the rapid expansion of UD has increased the demand for freshwater resources, which can exaggerate the water shortage in arid regions.

Reuse of PW can reduce freshwater usage and wastewater disposal, thus significantly enhancing the economic benefits and environmental sustainability of O&G production [16–19]. Currently, PW reuse is increasing within the O&G sector to support HF operations, and other potential beneficial uses outside the O&G sector have also been identified [10]. However, PW is considered one of the most complex aqueous mixtures [20]. Detailed characterization of the physicochemical and biological composition of PW is critical to monitor field operation and process, evaluate treatment efficiencies, assess potential risks on public health and environment during PW reuse, and inform management practices and regulatory compliance.

Despite numerous studies on PW analysis, there are no standardized methods approved by the United States Environmental Protection Agency (EPA) [21]. In the Code of Federal Regulations, title 40 (40 CFR), Part 136 (Guidelines Establishing Test Procedures for the Analysis of Pollutants), the EPA identified approved test methods for inorganic analytes (76 parameters), radioactive analytes (5 parameters), non-pesticide organic compounds (120 parameters), pesticides (70 parameters), biological indicators (8 parameters), and aquatic toxicity (4 parameters) [22]. The EPA has published these methods in "Selected Analytical Methods for Environmental Remediation and Recovery (SAM) 2017" [23]. The European Union (EU) has different water quality directives for different water bodies, such as 98/83/EC (drinking water), 80/68/EEC (groundwater), and 75/440/EEC (Surface water). However, these standard water quality analysis methods/directives usually only apply for freshwater (total dissolved solids, TDS < 1000 mg/L) or municipal and industrial wastewater. Since the TDS concentrations in PWs may range from less than 3000 mg/L to over 300,000 mg/L, these highly saline waters are difficult to analyze. Some of the traditional analytical methods do not work accurately for an intricate water matrix like PW. Danforth et al. reviewed 129 articles with detailed chemical analysis for 173 sources of PW collected from 27 locations in North America. They identified 1198 unique chemical constituents in PW, and only 290 (24%) could be quantified by the EPA-approved test methods. They also found that these studies are predominantly focused on the Marcellus Basin in Pennsylvania, even though the majority of produced water volume is generated in areas such as Texas, California, and New Mexico [8]. Thus, the development of suitable analytical methods for accurate PW characterization in complex water matrices is imperative.

This review aims to evaluate the commonly used analytical techniques for qualitative and quantitative analysis of PW. A tiered analytical approach is proposed to meet different operational and regulatory needs. For this critical review, we focused on the papers published after 2012 (and after 2016 for organic analysis) and reviewed 157 peer-reviewed publications and regulatory standard methods. PW compositions compiled from these sources are first presented and compared spatially and temporally to identify the challenges of PW characterization. The analytical techniques used in research papers are then compared with available regulatory standard methods from the EPA, including field sampling and sample preservation methods and pretreatment methods, basic water quality measurements, inorganic and organic analysis, radioactive measurements, and biological analysis. Based on the assessment of analytical tools, suitable analytical procedures are proposed to characterize PW samples. The review also identifies knowledge gaps and research needed to improve future PW characterization methods.

2. Water Composition

The physical and chemical properties of PW generally vary noticeably based on the geographic location, geologic formation, source oil, and chemical additives selected by a drilling company (spatial variation). These properties also depend on the time the PW samples are collected after well completion (temporal variation). The water constituents of the initial FW differ significantly from the PW [24]. Oetjen et al. examined the water chemistry of an HF site in the Niobrara formation (Colorado) throughout the flowback period and used principal component analysis to identify different stages in the flowback period [25]. For the well investigated, the majority of FW usually occurred in 1–2 days, the transition stage could last 6–21 days, and the PW stage generally started after 21 days [25].

Because PW contains a large portion of formation water, it generally contains native or geogenic constituents associated with the geological formation. In addition, many different chemical additives are injected into the wells during HF. Thus, the chemical constituents of PW often come from two sources: native constituents from the geologic formation and chemicals from fracturing fluids [5]. Native constituents often include salts, oil and grease, naturally occurring radioactive materials (e.g., radium, radon, strontium, uranium, and thorium), inorganic substances (e.g., ammonia and hydrogen sulfide), metals (e.g., aluminum, arsenic, barium, sodium, potassium, iron, and zinc), volatile gases (e.g., CH_4 and CO_2), and hydrocarbons (e.g., alkylbenzenes and polycyclic aromatic hydrocarbons (PAHs) and phenols) [5]. Another essential constituent in PW is bacteria, which are ubiquitous in the environment. They can originate from the geological formation itself, the source water used to create the fracturing fluid, and the associated infrastructure. [26].

Table S1 in Supporting Information (SI) summarizes the general physicochemical parameters of PW quality from primary UD plays in the U.S. PW from Bakken, Barnett, and the Permian contains high TDS (average >140 g/L) and relatively low dissolved organic carbon (DOC, average ~100 mg/L). In contrast, the water from Niobrara shows opposite trends, lower TDS (~40 g/L) and higher DOC (~1000 mg/L). The composition of PW also changes significantly during the well production process. Figure S1 in Support Information shows the temporal change of PW quality in Marcellus formation in Pennsylvania and Niobrara formation in Colorado. Because of the higher proportion of formation brine, PW typically has considerably higher TDS concentrations than FW. However, FW can have higher organics due to organic additives in fracturing fluid [24,27,28].

The primary types of organic additives used in HF fluid have been reported [6,7,10] and are publicly available on the FracFocus website. Table 1 summarizes the commonly used chemical additives, including acid, biocide, breaker, clay stabilizer, corrosion inhibitor, crosslinker, friction reducer, gelling agent, iron control, non-emulsifier, pH adjustment agent, scale inhibitor, and surfactant. It is important to note that the total chemical additives in HF fluid are only up to 0.5–1%. However, these unique chemicals can be used to track the PW related to UD. Although significant efforts have been made to disclose chemical additives used during HF, some additives are only identified by their generic names or are protected by proprietary claims such that sufficient information to identify the chemical structure is not made publicly available. To date, the identities of a fraction of the chemicals used in fracturing operations remain unknown. Identifying and detecting these additives present a particular challenge for environmental monitoring.

Table 1. Common groups of chemical additives in HF fluid.

Additive	**Example of Chemical**	**Chemical Purpose**	**Concentration (of Total Fluid)**
Acid	Hydrochloric acid	Help dissolve minerals and initiate cracks in the rock	0.07–0.15%
Biocide	Glutaraldehyde	Eliminate bacteria that produce corrosive by-products	0.075–0.06%
Breaker	Ammonium persulfate	Allow a delayed break down of the gel	0.02–0.06%
Clay stabilizer	Choline chloride	Prevent clays from swelling or shifting	0.05–0.2%
Corrosion inhibitor	Methanol	Product stabilizer and/or winterizing agent	0.002–0.004%
Cross-linker	Petroleum distillate	Carrier fluid for borate or zirconate crosslinker	0.007–0.032%
Friction reducer	Polyacrylamide	"Slick", the water to minimize friction	0.05–0.07%
Gelling agent	Guar gum	Thicken water to suspend the sand	0.05–0.5%
Iron control	Citric acid	Prevent precipitation of metal oxides	0.006 −0.011%
Non-emulsifier	Lauryl sulfate	Prevent the formation of emulsions in the fracture fluid	
pH adjusting agent	Sodium hydroxide	Adjust the pH of the fluid to maintain the effectiveness of other components, such as crosslinkers	0.01–0.011%
Scale inhibitor	Sodium polycarboxylate	Prevent scale deposits in the pipe	0.075–0.12%
Surfactant	Lauryl sulfate	Increase the viscosity of the fracture fluid	0.05–0.1%

Note: Data sources: references [5,6] and information from FracFocus, 2020 (https://fracfocus.org/chemical-use/what-chemicals-are-used).

The bacteria in PW also vary with the location, overall well condition, and well age [29]. Numerous studies investigated the microbial community composition and functional potential across the PW because of bacteria, such as sulfate-reducing bacteria, iron-oxidizing bacteria, and acid-producing bacteria. These bacteria in PW can cause corrosion, fouling, and sulfide release, resulting in production interruptions, harmful environmental consequences, and potential risks to public health [30,31]. Generally, the bacterial communities in the pre-fracturing (groundwater or surface water) fluid are mostly from the classes of *Alpha-proteobacteria* and *Gamma-proteobacteria*. During HF, the aerobic surface water microbial community rapidly transits to the halophilic, fermentative, and anaerobic microbial community in PW because of the extreme conditions, such as high salinity. Salinity is a major factor controlling the bacterial community composition in an aquatic environment [32]. Despite the use of biocides in HF fluid, high bacterial cell counts have been identified in PW. In the Marcellus and Bakken Shale, the microbial abundance in PW varied between 10^1 and 10^4 16 S rRNA gene copies/mL while it was 10^6 to 10^7 copies of the 16 S rRNA gene/mL in the pre-fracturing fluids [26,33,34]. The reasons may be related to the low efficiency/dosage of biocides, and the recycling of PW for HF may cause the enrichment and growth of bacteria [26]. The dominant bacterial classes found in PW include *Alpha-proteobacteria*, *Beta-proteobacteria*, *Epsilon-proteobacteria*, *Gamma-proteobacteria*, *Clostridia*, *Bacilli*, *Synergistetes*, *Bacteroides*, and *Flavobacteria* [26,35]. The dominant bacterial order found in Marcellus Shale was *Halanaerobiales*, followed by *Clostridiales*, *Campylobacterales*, *Rhodobacterales,* and *Bacillales* [36]. Hull et al. investigated the bacterial community succession in the Denver-Julesburg (DJ) Basin (Niobrara shale formation, Colorado). They found that class *Gamma-proteobacteria* was dominant in groundwater (62%). However, the PW sample collected four days after flowback began was dominated by *Thermoanaerobacter* of *Clostridia* (97%), and its relative abundance decreased as *Thermovigra of Synergistia* and *Thermotoga* of *Thermotogae* started to increase at day 55 and 80, respectively. After 220 days, the dominant classes in PW were *Clostridia* (50%) and *Thermotogae* (40%) [37].

As shown by the data and discussion above, PW appears to exhibit a considerable variation in general physicochemical and biological properties. Better characterization of the constituents in PW by location and time is critical to monitor process operation, choose proper management and treatment methods, and guide beneficial PW uses.

3. Tiered Analytical Approach

Figure 1 illustrates the life cycle of produced water management from HF, production, gathering and transportation, treatment, storage (e.g., above-ground storage tank (AST) and recycle impoundments), sourcing and blending, chemical additions, reuse for

HF, advanced treatment (e.g., desalination) for beneficial applications, to waste disposal (e.g., solids to landfill, concentrate to underground injection control (UIC) disposal wells). Analytical testing is necessary throughout all stages and levels of the water cycle. The analytical data will be used to characterize influent streams, model potential technology applicability, monitor process performance, and assess environmental and public health risks of treated water applications and treatment waste streams. There is a need for both rapid testing techniques to support process quality assurance (QA) as well as more detailed characterization testing for proper disposal, treatment, and reuse. As a result, we propose a multi-tiered approach and classify the PW analysis into four main tiers (Table 2) based on the needs to meet different purposes of analysis, cost, complexity, and turn-around time (Figure 2).

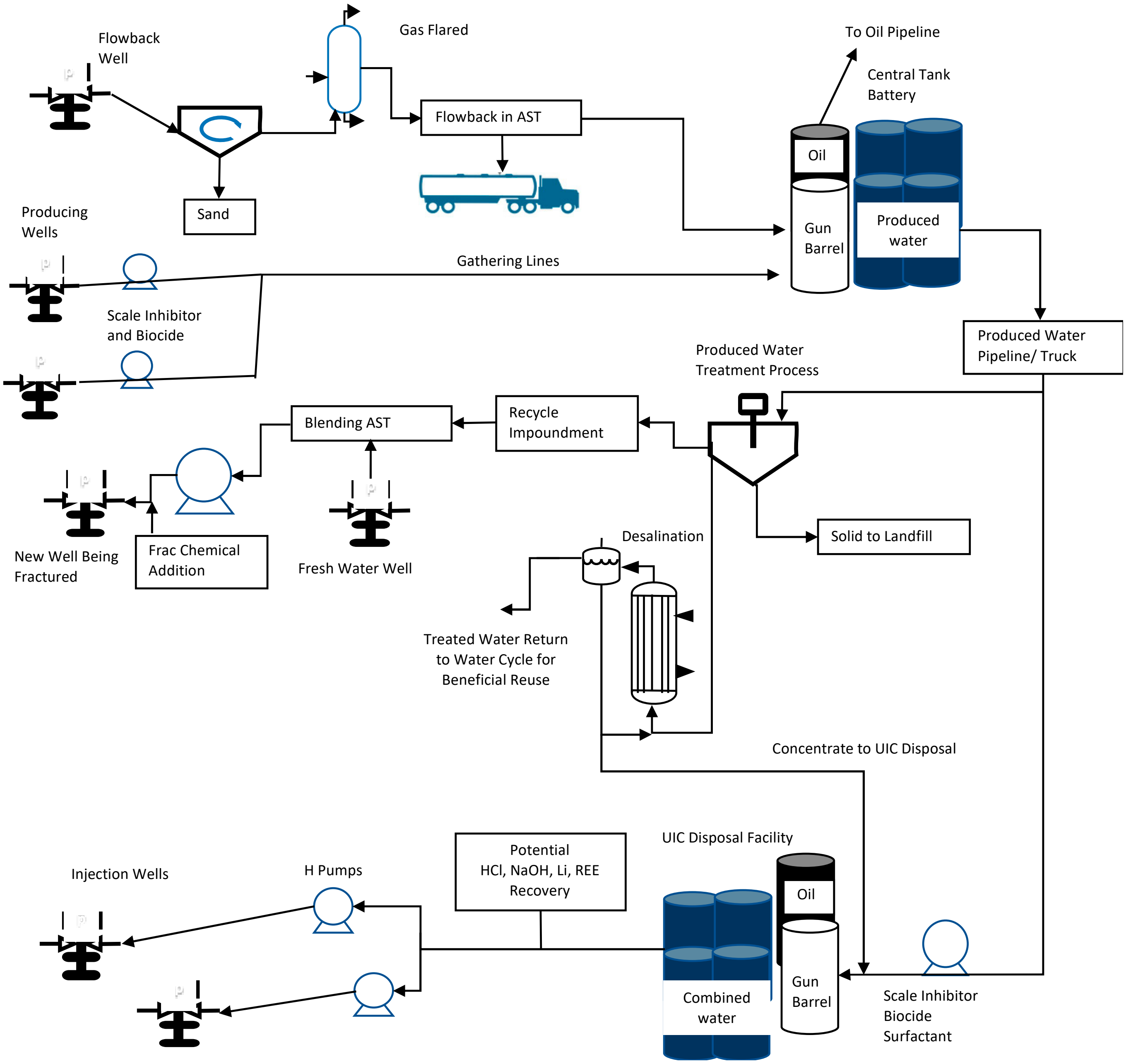

Figure 1. Schematic diagram of produced water production, transportation, treatment, reuse for hydraulic fracturing, advanced treatment for beneficial uses, and disposal. AST: Above surface storage tank; UIC: underground injection control.

Table 2. Multi-tiered approach for produced water characterization.

Level	Use	Description	Parameters	Frequency
Tier 1	Continuous monitoring, bulk testing, KPI rapid analysis, process control	In-Line Sensors Field Parameters Filter Analysis	Flow, TSS, TDS, TOC, pH, ORP, iron, H_2S, TPH, level sensing, carbonate	Realtime, continuous, and routine
Tier 2	Detailed characterization, routine monitoring, and Tier 1 data verification	Conventional Lab Testing	Wet chemistry, ICP-OES, ICP-MS, GC, GC-MS, HPLC	Baseline, quarterly, when experiencing data excursions in Tier1. Proving up treatment efficacy and reliability, beneficial reuse investigation
Tier 3	NPDES discharge compliance, modeling treatment technology; Waste disposal profile generation; Risk assessment and data capture for fate/transport modeling.	Unconventional Lab Testing; WET Testing Leachate Testing Bio-mobility and accumulation testing	LC-MS, Gamma Spec, High Res GC-MS; Acute and chronic toxicity TCLP, SPLP, LEAF testing of residual waste Tier 1,2,4 analysis of treated effluent on soil, plant, tissue samples	When evaluating technology and management processes. As per permit/regulatory agency
Tier 4	Source apportionment, fingerprinting	SEM/EDX, XRD, FEEM, biomarker analysis, isotopic analysis		Evaluating technology and management process. Basic research for method development. Event response. Beneficial reuse investigations.

Note: TSS: total suspended solids; TDS: total dissolved solids; TOC: total organic carbon; ORP: oxidation reduction potential; TPH: total petroleum hydrocarbon; ICP-OES: inductively coupled plasma-optical emission spectroscopy; ICP-MS: inductively coupled plasma-mass spectroscopy; GC-MS: gas chromatography-mass spectroscopy; HPLC: high-performance liquid chromatography; LC-MS: liquid chromatography–mass spectroscopy; TCLP: toxicity characteristic leaching procedure; SPLP: synthetic precipitation leaching procedure; LEAF: Leaching Environmental Assessment Framework; SEM/EDX: scanning electron microscopy/energy dispersive X-ray spectroscopy; XRD: X-ray diffraction; FEEM: Fluorescence excitation-emission matrix.

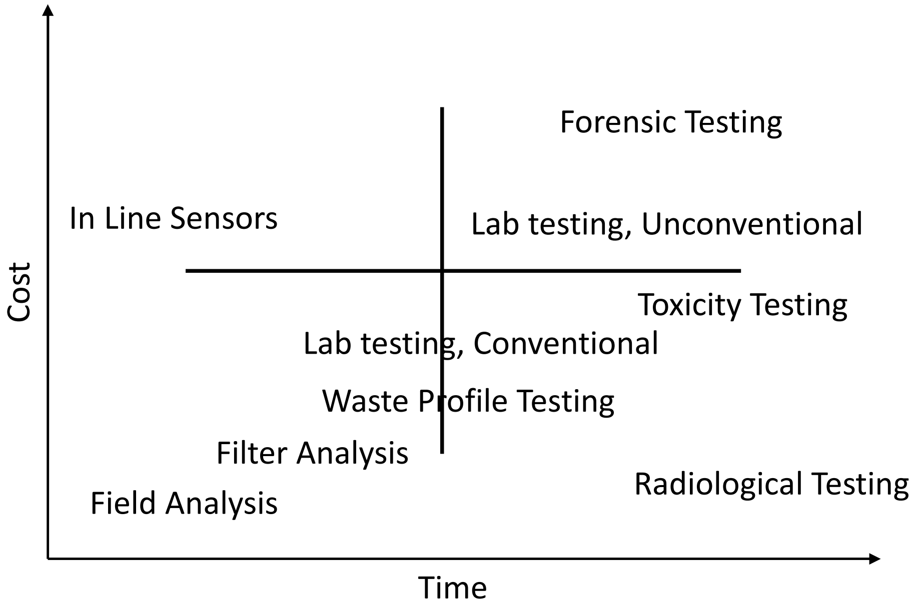

Figure 2. The cost and turnaround time of produced water analysis.

Tier 1 analysis involves using in-line sensors, field parameters, and onsite testing for real-time, continuous monitoring, and routine process control. An efficient indicator system could be established with key performance indicators (KPIs) to support the control of operations and processes within certain limits and for internal and external reporting. The KPIs need to be formulated from different perspectives depending on the operation and process. They may include flow, total suspended solids (TSS), conductivity/TDS, pH, temperature, oxidation-reduction potential (ORP), iron, H_2S, alkalinity, total organic carbon (TOC), suspended and colloidal particles, and total petroleum hydrocarbon (TPH). One challenge for Tier 1 analysis is to maintain the accuracy of these in-line sensors, which are installed under harsh conditions. Routine calibration/replacement is required to ensure high-quality data. Another challenge is the data analysis/classification from these in-line sensors. Advanced statistical methods, software tools, machine learning, and connected digital systems are required for data processing, failure analysis, predicting performance, and operation control. An excursion from historical control limits detected by in-line Tier 1 sensors will trigger grab sample capture and Tiers 2 to 4 characterization analysis. Tiers 2 to 4 focus on detailed characterization, routine monitoring and Tier 1 data verification, National Pollutant Discharge Elimination System (NPDES) permit compliance, evaluating treatment technology and management processes, and as per permit/regulatory agency for beneficial uses, or event responses. Conventional and unconventional lab testing, Whole Effluent Toxicity (WET) testing, leachate testing, and fingerprint testing will be conducted using advanced analytical tools. For example, ion chromatography (IC), inductively coupled plasma mass spectrometry (ICP-MS), and inductively coupled plasma optical emission spectrometry (ICP-OES) analysis can provide elemental and ionic makeup of TDS. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) can provide information about particle size, morphology, and crystal structure. A variety of techniques can be employed to characterize the same common parameters at different levels.

4. Field Sampling, Preservation, and Sample Pretreatment

4.1. Field Sampling and Preservation

Environment sampling is a crucial process to ensure the certainty of analytical results. Several important aspects need to be considered for the field sampling process, including sampling points and locations, analyte selection and the number of samples, field measurements and sampling log, containers and preservation, quality assessment samples, and other related information.

Sampling points and locations should represent the PW management process and have to be consistent to ensure that the results from a different period of measurements are comparable. Common sampling points include wellhead, oil-water separator, storage tank, and points before and after treatment, as shown in Figure 3. Several parameters should be measured during the sampling process when each sample is collected to estimate the sample variation and guide sample preservation methods, such as temperature, pH, free chlorine residual, gross density, and conductivity. Free chlorine measurements will be used to guide the sample preservation.

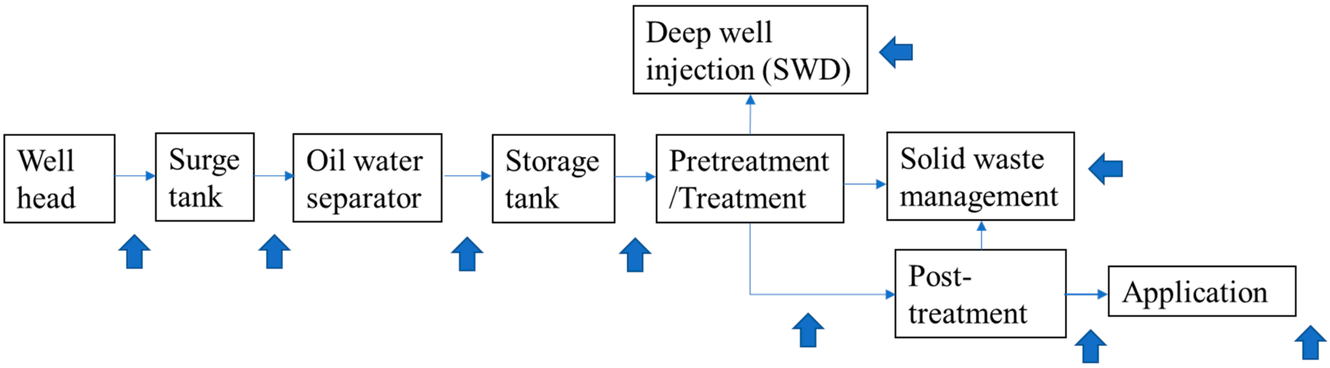

Figure 3. Example of water sampling and analysis locations and tiers.

The details of different sample containers and preservation methods for the target analytes are discussed in Support Information. Based on the methods generally used by commercial labs under the EPA guidance, Table S2 summarizes the analytical methods, containers, preservations, and holding times for PW analysis of wet chemistry, anions, total metals, organics, radioactive, and WET testing.

4.2. Sample Preparation and Pretreatment

Sample preparation is essential for PW analysis. It has several goals: (1) to concentrate or dilute target analytes to meet the capability of analytical instrumentation; (2) to remove materials in the matrix that might interfere with the chromatographic separation, ionization, or detection of target analytes. For inorganic analysis, these goals are usually met by removing particles and diluting the sample to meet instrument performance. For organic compound analyses, removing inorganic ions in PW while retaining specific organics in the final solution is often required. The EPA's SW-846 compendium consists of over 200 analytical methods for sampling and analyzing waste and other matrices. It includes the 3000 series for inorganic sample preparation, 3500 series for organic sample extraction, and 3600 series for organic extract cleanup. A variety of sample preparation methods suitable for PW samples are discussed in the following sections.

4.2.1. Liquid-Liquid Extraction and Solid-Liquid Extraction

Liquid-liquid extraction (LLE) is currently the most widely used organic compounds extraction method for PW, especially to extract semi-volatile compounds for gas chromatography (GC) analysis, due to its simplicity and ease of method development [38,39]. It has also been widely used in EPA-approved methods to extract GC amenable organic compounds in water samples (solid-liquid extraction is used for solid samples, discussed below), such as in EPA Method 625. It usually uses an organic solvent to extract non-polar compounds from PW, targeted non-polar analyte can be collected from the organic solvent, and the hydrophilic analyte is left in the PW matrix. The most widely used organic solvent for LLE in PW analysis is dichloromethane (DCM), the recommended solvent in the EPA methods. LLE has several limitations when applied to treat PW samples. First, it has low selectivity; the analyte is often extracted with other compounds, which increases background noise. When a large number of interferences exist in the extract, a cleanup step is required.

The cleanup procedure of the silica gel column is recommended in the EPA Method 610. Second, when LLE is used to collect water-soluble compounds, dilution is often required to deal with the high TDS in the water matrix, which increases the minimum detection limit (MDL) for the analyte. Finally, the most considerable disadvantage of this method is the formation of emulsions during extraction, especially for PW samples, which contain large amounts of surfactant-like compounds. The surfactant creates a mid-phase, making the clean collection of one phase difficult [40,41]. However, emulsions can be reduced by using a continuous extractor as described in the EPA Method 625 or adding salt into the solution, as salt changes the capacity of the aqueous phase, driving slightly soluble compounds into the organic phase. Salt-assisted LLE is more widely used to extract organics from PW samples [25,42].

Solid–liquid extraction (SLE) can extract analytes from contaminated soil or other solids related to O&G activities. Organic solvent and solid are usually mixed under ultrasonic, which increases extraction yield and decreases extraction time (EPA Method 3550C). The mixture is then centrifuged, and the supernatant is collected for further treatment. Accelerated solvent extraction (ASE) is an SLE method for extracting various chemicals from a complex solid or semisolid sample matrix. It uses high temperature and pressure to accelerate the extraction process further, increase extraction yield, and decrease the amount of solvent required [43]. SLE and ASE have been used to extract organics adsorbed on particulates in PW for a comprehensive analysis of the total organics in PW samples [13]. Soxhlet extraction (EPA Method 3540C) is also a widely used method for extracting nonvolatile and semivolatile organic compounds from solids such as soils, sludges, and wastes. It uses a Soxhlet extractor to ensure intimate contact of the sample matrix with the extraction solvent, which improves extraction efficiency to extract targeted compounds from solid matrices.

4.2.2. Solid-Phase Extraction

Solid-phase extraction (SPE) is a powerful and widely used extraction technique that offers high selectivity, flexibility, and automation. The EPA Method 3535A is a procedure for isolating target organic analytes from aqueous samples using SPE media. SPE has been widely applied to concentrate and purify analytes from different water matrices, including wastewater and PW [25,31,44]. It can easily remove the interferences (such as salt, insoluble material, and unwanted organics) in PW without the formation of emulsion and allow concentrating the analyte during sample preparation to decrease the MDL, which is crucial to meet environmental regulations. It can also be used to collect different groups of compounds from one SPE column based on their polarity. Sorensen et al. collected nonpolar compounds from the SPE column first by using n-hexane followed by DCM, and then the polar part was collected by using methanol (5–20%) in DCM as an elution solvent [45]. Some researchers also used SPE to remove hydrophobic compounds in samples for IC analysis [46]. One disadvantage of this technique is that it requires more knowledge about the targeted analytes than other approaches. It requires more effort to find suitable sorbents and solvents when dealing with an unknown sample. Furthermore, the cost of the SPE is higher than that of other techniques [40]. Table S3 in Support Information summarizes the SPE cartridges used in literature for organic analysis.

Solid-phase microextraction (SPME) is an SPE technique that uses a fiber coated with an adsorbent material to extract analytes from the liquid or gas phase. After extraction, the SPME fiber is transferred to the injection port of separating instruments, such as GC, LC, or mass spectroscopy (MS), where desorption of the analyte takes place. SPME has several advantages, such as (1) it does not require a solvent and purge-and-trap instrument (discussed in the following section), which saves time and money; (2) it can be highly selective based on the fiber and adsorbent used for the analytes [38,40]. Khan et al. used SPME to pretreat PW samples from the Permian Basin and successfully characterized 1400 compounds, including benzene, toluene, ethylbenzene, xylenes (BTEX), alkanes, and alkylbenzenes [20]. Almaraz et al. used a polydimethylsiloxane/divinylbenzene (PDMS/DVB) fiber to extract five different iodinated organic compounds (chloroiodomethane, diiodomethane, triiodomethane, chlorodiiodomethane, and bromodiiodomethane) from PW. The extracted compounds were then analyzed by headspace gas chromatography-mass spectrometry (GC-MS). The fiber material, extraction temperature and time, and desorption time were also evaluated [47]. Redman et al. also used SPME for quantitative analysis of nonpolar and organic acids in PW, which can then be linked to toxicity prediction [48].

Fabric phase sorptive extraction (FPSE) is a new type of SPE, similar to SPME [49]. It uses small squares of cellulosic (or other) fabric coated with an ultrathin sol-gel to sorb and extract analytes directly from aqueous environmental water samples. The analyte is then eluted from the fabric piece by a small volume of extraction solvent and injected into a chromatography system [50]. FPSE offers several advantages of short extraction times, minimal solvent use, and the ability to reuse coated fabric pieces after minimal cleaning. FPSE has been successfully applied to drinking water, surface water, wastewater, and biological samples; however, there is no publication using it for PW analysis [51].

4.2.3. Other Methods

Purge and trap is suitable for GC analysis. It is widely used in EPA-approved methods to extract organics from water and wastewater, such as the EPA Method 624.1. The sample is first placed in a sealed vessel; inert gas is purged into the sample to cause volatile compounds to be swept out. The gas with volatile compounds is passed through an adsorbent trap, where the volatiles are retained. The final step is to desorb the volatiles by heating the trap and using GC carrier gas to inject them into a GC instrument.

Derivatization is a useful sample preparation tool for organic analysis. It converts a specific compound into a product of a similar structure that is more suitable for analysis. A chromophore can be added to a compound to enable its detection in UV-Vis spectrophotometry. A polar or nonpolar group can be used to adjust the retention time of a compound in GC or LC, thus enhancing the separation efficiency. Derivatization can also be used to improve ionization efficiency for poorly ionizable compounds in MS [52]. Derivatization with 2, 4- dinitrophenylhydrazine is the recommended approach to analyze aldehydes, such as glutaraldehyde and formaldehyde; both are used as biocides in HF fluids [53,54]. Sorensen et al. used N,O-bis(trimethylsilyl)trifluoroacetamide (BSFTA) to derivatize all the acidified organic compounds extracted from PW for nontarget analysis, and BSFTA is used to protect the labile groups such as hydroxyl in the target analytes [45].

In summary, proper sampling and preservation are vital to analyzing the chemical constituents accurately in PW. Due to the complex PW matrix, sometimes multiple sample pretreatments are required to collect the target analytes. For example, LLE can be used as a first step to extract all the organics from liquid samples, and then the extract is further cleaned by SPE [13]. Alternatively, PW samples can be first acidified (HCl, $pH < 2$) to prevent degradation, followed by LLE (DCM) to extract all the organics from the water samples. The extract can then be further cleaned and separated by SPE for characterization by different analytical methods [45].

5. Bulk Measurements and Basic Water Quality Parameters

Bulk measurements are essential for any water analysis because they are quick and cost-effective and provide overall information about the water matrix. The informative bulk measurements include pH, conductivity, temperature, alkalinity, salinity, total suspended and dissolved solids (TSS and TDS), total organic carbon (TOC)/dissolved organic carbon (DOC), total nitrogen (TN), total petroleum hydrocarbons (TPH), oxidation-reduction potential (ORP), and others [55]. These basic parameters are valuable for monitoring well operation and guiding subsequent detailed analysis. Some industries use these parameters as process control, only performing a more detailed analysis when fluctuation is observed [56]. These measurements can be performed on-site with probes/sensors or in the lab with a relatively simple instrument. Currently, there are commercial probes available for on-site measurements. For example, YSI Professional Plus multi-parametric probe can be used to measure temperature, dissolved oxygen, conductivity, TDS, salinity, pH, turbidity, and ORP [30]. Please refer to SI for detailed discussions on basic PW quality analysis. Table S1 includes some measurement results of the typical water quality parameters from different PW sources.

6. Organic Analysis

As discussed in Section 2, dissolved organic matter (DOM) in PW usually derives from a combination of native hydrocarbons (e.g., BTEX, PAHs, phenols, humic and fulvic acids), chemical additives (e.g., biocides and guar gum), and the transformation products under extreme conditions in the well (high temperature and pressure). Synthetic organic compounds from source water for well operation have also sometimes been detected. Alcohols (made up mostly of methanol) are the most abundant organic compound group in PW (approximately 91% of total volatile organic concentration) [57]. The organic constituents vary widely during the lifetime of the well operation, especially during the early stage of well drilling. Sun et al. investigated the organic content in PW in the Duvernay formation (Alberta, Canada). They found that most organic compounds declined in abundance over the first nine days of flowback, except certain kinds of compounds such as phthalate diesters and hydroxyquinoline, which were still observed on Day 30 [58].

Bulk measurements such as DOC, COD, and ultraviolet absorbance at 254 nm ($UV_{254\ nm}$) are convenient techniques to estimate the total DOM in aqueous samples. There are also several advanced analytical techniques for characterizing and quantifying DOM, including gas chromatography coupled with flame ionization detector (GC-FID), thermal conductivity detector (GC-TCD) or mass spectrometry (GC-MS), and liquid chromatography (LC) coupled with UV-diode array detector (LC-UV), organic carbon detection (LC-OCD) or mass spectrometry (LC-MS). GC-based methods are extensively used to analyze volatile and semivolatile organic compounds (VOCs and SVOCs), including natural gas constituents (methane and ethane), BTEX, diesel range semivolatile organics with detailed discussion in Section 6.2. LC-based techniques are more suitable for non-volatile organic compounds such as surfactants, fatty amines, high molecular weight ionic polyacrylamide friction reducers with detailed discussion in Section 6.3 [21].

One of the significant challenges to analyze organic compounds in PW is the lack of standardized and validated methods. There are EPA-approved standard methods for domestic and industrial wastewater, and they may be suitable for some PW analyses. For example, the EPA Method 610/Method 8275A/8270 SIM for determination of PAHs, which exist in PW, by both HPLC-UV and GC-FID; the EPA Method 553 for determination of Benzidines and Nitrogen-containing pesticides (used as chemical additives in HF fluids) in water by LLE or LSE and HPLC-MS are all plausible methods. However, they have not been validated to handle the PW matrix, which can be four times saltier than seawater. The EPA approved methods target specific or a series of known organic compounds, while numerous unknown organic compounds may exist in PW, which requires the development of new methods or modification of existing methods. The identification and quantification of individual organic compounds among the complex matrix of PW require various sample preparations (Section 4) and advanced analytical techniques [59].

6.1. Mass Spectrometry, Tandem Mass Spectrometry, and High-Resolution Mass Spectrometry

Mass spectrometry (MS) is the most potent detector to characterize complex fluids and has been extensively used for PW analysis. It can provide qualitative and quantitative information about the analytes with the help of standards or mass spectral libraries. MS identifies charged analytes that are produced by ionization through a mass analyzer. Different ionization methods and mass analyzers provide different benefits and analytical power.

There are several ionization methods, including electron ionization (EI), chemical ionization (CI), electrospray ionization (ESI), atmospheric pressure chemical ionization (APCI), and matrix-assisted laser desorption/ionization (MALDI). EI is the most common ionization method for GC analysis. Numerous studies used EI to investigate the VOCs and SVOCs in PW [60]. Because EI is a hard ionization method that breaks almost all the parent ions into fragments and leaves a small amount or no parent ion, it is difficult to identify the parent ion without standards or a library of MS fragmentation spectra [61]. On the other hand, the EI spectra are very reproducible when the "standard" EI settings are used, regardless of the equipment. Thus, substantial effort has been devoted to developing GC-MS libraries with EI using standardized conditions. Today, GC-(EI)-MS libraries contain hundreds of thousands of compounds for researchers to identify unknown compounds, such as the Wiley database with ~600,000 compounds and the NIST/EPA/NIH mass spectral library for EI-spectra containing ~267,376 compounds [62].

CI is a soft ionization method that does not break the parent ions into fragments; thus, more parent ions will remain. This property may help identify unknown compounds. However, to date, it has not been used in PW analysis. ESI and APCI are both soft ionization methods to couple LC and MS. ESI is the most commonly used ionization method for LC-MS, and there is no literature reported using APCI for PW analysis. While EI generates radical cations, ESI usually generates protonated/deprotonated or adduct ions (Na^+-, K^+-), which depends on the characteristics of the analyte and the experimental conditions. There are three major issues with ESI for PW analysis. First, some compounds (such as surfactant) will interfere with the ionization of other compounds. Second, a high concentration of sodium (and other ions) in PW usually generates unpredictable adducts during ionization, hindering quantification. Third, LC-(ESI)-MS libraries are at a much smaller scale compared to GC-(EI)-MS libraries, due to lack of standardization for MS conditions and differences in spectra generated on different brands and types of mass spectrometers and also because it was developed more recently [41,62]. MALDI is a soft ionization method that uses a laser energy absorbing matrix to create ions from large molecules with minimal fragmentation. It has been widely applied to the analysis of biomolecules and large organic molecules and has also been applied to identify microorganisms in PW, as discussed in Section 7 [30,63].

Standard mass analyzers include the magnetic sector, quadrupole (Q), ion trap, and time of flight (ToF) mass analyzer. The general characters of each mass analyzer are summarized in Table 3. The quadrupole is the most commonly used mass analyzer for PW samples because of its availability to researchers and good performance [41]. It consists of four precisely matched parallel metal rods, and a high-frequency oscillating electric field is created in these rods. By adjusting the electric field, only selected ions can pass through the geometry of quadruple rods. This approach has many advantages, including high reproducibility, easy to couple with GC or LC, and relatively low maintenance. A significant disadvantage of this detector is its low resolution, which means it cannot separate compounds with close molecular mass and leads to false-positive identifications in mass spectral libraries. The situation worsens when a large variety of compounds exist in a PW sample. ToF is the second most broadly used mass analyzer for PW samples; its performance is better than Quadrupole (Table 3). It separates the ions by the times they needed to travel through a long field-free flight tube; larger mass ions move slower and need more time to reach the detector. It is usually coupled with LC to identify surfactants in PW. It has been crucial in identifying polypropylene glycols, polyethylene glycols (PEGs), and ethoxylated surfactants [64,65]. Ion trap mass analyzers use oscillating electric fields or radiofrequency to trap and separate ions. They can achieve very high resolution but are limited in their capacity to trap ions. The standard configurations include the Quadrupole ion trap, Orbitrap, and Fourier-transform ion cyclotron resonance mass spectrometry (FT-ICR-MS). Orbitrap and FT-ICR have very high resolution and become more critical and popular for PW analysis.

Table 3. Comparison of standard mass analyzers [66,67].

	Magnetic Sector	Quadrupole	Quadrupole Ion Trap	Time of Flight (ToF)	Orbitrap	FT-ICR
Mass range (Da)	15000	4000	4000	Unlimited	$>10^4$	$>10^4$
Resolving power	10^2–10^5	4000	10^3–10^4	15,000	$>10^5$	$>10^6$
Mass accuracy (ppm)	1–5	100	50–100	5–50	2–5	1–5
Scan speed (Hz)	0.1–20	1–20	1–30	10^1–10^6	10^{-1}–10^1	10^{-2}–10^1
MS/MS	Excellent	Great	Great	Great	Great	Great
Cost	$$$$	$	$	$$–$$$	$$$	$$$$

Tandem mass spectrometry (MS/MS) requires two or more mass analyzers coupled in series that are separated by a collision cell to provide fragmentation of selected ions (parent ions) [66]. Conventionally, this technique is best applied for quantitative analysis of specific ions or compounds. It can also provide structural information and further confirm the identity of the unknowns. Common MS/MS include ion-trap, triple-quadrupole (QQQ), and quadrupole-time-of-flight (Q-ToF). When a low-resolution mass analyzer is used, it only acquires nominal masses and can hardly be used for nontarget analysis. However, when paired with high-resolution mass spectrometry (HRMS), MS/MS can provide crucial information to elucidate the elemental composition and structure of the compounds in the sample matrix [41].

HRMS has been successfully applied for discovering unknown contaminants in environmental (and other) samples and will continue to play a key role in identifying new unknown compounds in PW because of its remarkable ability to provide rich structure information that enables compound-specific determinations [68,69]. Currently, the most widely used HRMS/MS includes Q-ToF, Orbitrap mass analyzer, and FT-ICR-MS. However, confidence in the HRMS-based identifications varies between studies and compounds because it is not always possible to find the standards or confirm them via complementary methods. Thus, Schymanski et al. proposed identification confidence levels in HRMS, which have been widely accepted by researchers. The confidence levels are classified into (1) Level 1 (highest confidence), confirmed structure, where the proposed structure can be confirmed by appropriate measurement of a reference standard with MS, MS/MS, and retention time matching; (2) Level 2, probable structure, Level 2a involves unambiguous spectrum-structure matching with literature or library spectrum data, while Level 2b applies when no standard or literature information is available for confirmation, but no other structure fits the experiment information; (3) Level 3, tentative candidate, where evidence exists for possible structure(s), but the exact structure cannot be confirmed; (4) Level 4, unequivocal molecular formula, where a formula can be unambiguously assigned based on the spectral information, but no possible structure(s) can be proposed; (5) Level 5 (lowest confidence), exact mass (m/z), where exact mass can be measured but lack information to assign even a formula [70].

Confidence Level 2 (library matching) is a more convenient method to identify unknown compounds than Level 1 due to the problem of the standards that are not always available, and sometimes the standards can be costly [13,58]. As a result, many separate open and semi-open mass spectrometry databases exist, such as Metlin, MassBank, European MassBank, MassBank of North America, the Global Natural Products Social Molecular Networking (GNPS), and ChemSpider. Two commercial MS/MS libraries, NIST and Wiley, are also available. The NIST20 library database released in June 2020 contains MS/MS spectra for 31,000 compounds, 186,000 precursor Ions, and 1.3 million spectra [71], while the Wiley's MSforID database contains MS/MS spectra for >1200 compounds [51].

Some disadvantages limit the application of MS in PW analysis. The most important reasons are the high capital and maintenance costs, the requirement for well-trained researchers, the lengthy sample analysis and data processing time, and the expensive MS database and standards discussed above. Besides, this technique has some intrinsic drawbacks for PW analysis. First, the resolution of standard mass analyzers decreases with the increases of ions, which means it is essential to separate target analytes from background ions and matrix in PW prior to injection. Thus, significant sample pretreatment (cleanup) is always required in order to achieve acceptable results. Second, the ionization efficiency varies between different compounds and is strongly affected by the sample matrix, especially for ESI. Inorganic or organic matrix constituents that co-eluted with target analytes from LC may enhance or suppress the ionization of the target analytes. The complex matrix chemistry can affect how a target analyte is ionized. For example, an analyte may form a disproportionate amount of sodium adducts $[M + Na]^+$ during ESI when present in saline PW samples instead of forming protonated adducts $[M + H]^+$ when present in clean water matrix, especially for some additives because of their oxygen-rich structures [54]. Sodium adducts pose a problem for typical quantification methods because they do not fragment and protonate adducts [65,72]. This problem limits the application of existing LC-ESI-MS methods for quantifying organics in untreated PW samples, especially the PW sample matrices change over location and time; thus, standard methods are even harder to establish.

6.2. Volatile and Semi-Volatile Organic Analysis

Volatile organic compounds (VOCs) are organic chemicals with a high vapor pressure at ordinary room temperature (low boiling point), while non-volatile organic compounds, in contrast, have a low vapor pressure. Semi-volatile compounds (SVOCs) are the compounds with a vapor pressure between VOCs and non-volatile compounds [73]. In general, more effort has been focused on the analysis of VOCs and SVOCs in PW, which are more amenable to GC for the analysis of non-volatile compounds. Headspace gas chromatography (HSGC) is a convenient method to analyze VOCs such as methanol and ethanol in PW because it reduces the required sample preparation and minimizes matrix interferences. HSGC injects headspace gas, from the top of a sealed container containing a liquid or solid brought to equilibrium, directly into a GC column for separation and analysis. HSGC can also analyze VOCs in contaminated soil near shale basins for UD [74]. The VOCs and SVOCs can be analyzed by GC coupled with numerous types of detectors. One of the most commonly used non-selective detectors is the flame ionization detector (FID), which detects the organic compounds eluted from a separation column. GC-FID is used in the EPA Method 8015 to detect alcohols and organic acids [75]. It has been used in PW analysis to detect total organic matter amenable to GC [45]. However, FID and other non-selective detectors could not overcome the PW matrix issues. Thus, the selective detector, MS, is often required to couple with GC for PW characterization [41].

Besides the advantage of easy sample preparation, a large amount of mass spectrometry database and accessibility are other two reasons that have stimulated the application of GC-MS for VOCs and SVOCs analysis in PW, as discussed in Section 6.1 [20]. Comprehensive two-dimensional GC paired with ToF-MS (GC × GC-ToF-MS) with a higher resolution has also been used to identify a large number of volatile compounds in PW, including geogenic compounds, fluid additives, and potential transformation products [45,61]. GC × GC is a technique that generally separates the analytes by the boiling point on the first column and then by polarity on the second column. It is powerful enough to analyze very complex mixtures. However, very few studies have used it, likely due to high expense, need for qualified operators, and extensive data processing [55].

6.3. Non-Volatile Organic Analysis

Analysis of more polar and non-volatile organic compounds is challenging due to the complexity of the PW matrix of and the lack of knowledge of what compounds may be present in PW, especially for the transformation product analysis. For example, alkyl ethoxylates (AEOs) are usually used as surfactants in HF fluid, but they are rarely detected in PW. Recent studies proved that the AEOs transformed to PEGs through central cleavage of the ethoxylate chain from the alkyl group, making the detection of AEOs difficult without knowing its transformation product [76].

High-performance liquid chromatography–mass spectrometry (HPLC-MS) is a useful tool to analyze non-volatile compounds in PW. HPLC-MS has been used to identify compounds such as glutaraldehyde, amino ethoxylates, and propoxylates [53,69]. However, little research has been conducted to characterize PW or monitor groundwater located near UD sites by using HPLC-MS compared to the use of GC-MS [5]. This could be due to the high instrument cost, lack of libraries for MS data when using ESI-MS, and the complexity of PW matrix [21,60].

Luek et al. [60] reviewed 18 publications analyzing organic compounds in shale gas PW from 2009 to 2016 and found 14 publications used GC-based techniques, and only 4 used LC-based techniques; 1 publication used FT-ICR-MS, and Orbitrap was not reported. Table S4 summarizes 25 peer-reviewed publications analyzing organic compounds in shale gas PW from 2016 to date. In summary, 14 publications used LC-MS, while 13 used GC-based techniques (the overlap is because some publications used both techniques). This trend may be a result of advances in HRMS and ultra-HRMS, in addition to the concerns surrounding undisclosed proprietary chemicals used during HF and their transformation products during well production. Orbitrap (7 publications) and Q-ToF (7 publications) have become the dominant HRMS/MS analyzers because of their high resolution and relatively low price. In comparison, only two publications from the same group used FT-ICR-MS, likely due to its high cost despite the high resolution.

Nontarget analysis has become a more important tool to discover “unknown” chemicals in PW samples using HSMS/MS (Table S4). This approach requires no prior information about the unknown chemicals in PW. The unknown chemicals are defined as chemicals that have not been previously confirmed by reference standards or are not reported on suspects lists (such as in FracFocus). The basic procedure for nontarget analysis is first to collect the mass spectra of unknown chemicals, and data processing techniques are used to assign potential molecular formulas. Chemical structure identification is achieved by database spectra searching (confidence Level 2) or matching the MS/MS spectra and retention time with reference standards (confidence Level 1) [41]. One drawback of nontarget analysis is the possible bias that resulted from sample preparation. Because SPE is the most suitable pretreatment for LC-MS but requires prior knowledge about the analytes to optimize the procedure, more effort is required to analyze PW without losing useful information during comprehensive nontarget analysis.

Sitterley et al. used nontarget analysis and discovered amino-poly (ethylene glycol)s, amino-poly(ethylene glycol) carboxylates, and amino-poly(ethylene glycol) amines in PW samples from HF in the western United States [69]. These compounds were not listed in FracFocus reports and categorized as a proprietary surfactant blend. They first used HPLC-ultrahigh-definition Q-ToF MS for the sample analysis. The measured accurate mass from the total ion chromatogram peak was used to obtain a neutral formula with the MassHunter formula generator. After searching the ChemSpider database for this formula, a polyethylene glycol (PEG) structure containing a primary amine on one end and a hydroxyl group on the other end was given. They also noticed that the mass difference between the major peaks was the calculated exact mass of an ethylene oxide unit ($O\text{-}CH_2\text{-}CH_2$). Thus, they suspected the peaks were amino-PEGs with ethoxylated chains in the range of $n = 5$–8. The analytical standard (confidence level 1) of $C_{12}H_{27}NO_6$ (amino-PEG6) was obtained to verify their hypothesis. The putative amino-PEG6 in the PW sample and standards were run for MS/MS analysis. The chromatograms for each one has the same retention time and nearly identical MS/MS mass spectra. Kendrick mass defect was used to verify the series of amino-PEGs because compounds that only differ by the addition or subtraction of one or more ethylene oxide units are related by having the same Kendrick mass defect. Other series of compounds were discovered in the same way.

6.4. Three-Dimensional Excitation-Emission Matrix Fluorescence Spectroscopy

The techniques discussed above have similar disadvantages, such as high cost, the requirement of meticulous sample preparation, and the time required to obtain the results.

Quick and effective characterization and quantification of DOM is required to facilitate broad reuse of PW. Three-dimensional fluorescence spectroscopy is an alternative approach to characterize DOM in PW because most organic molecules, such as proteins and bacterial metabolites, have fluorescent emission characteristics. 3D fluorescence produces an excitation-emission matrix (EEM) depending on the chemical structure and functional groups of DOM in the sample. Different groups of organics can be separated based on their intensity or location in the EEMs. 3D fluorescence measurements are quick and comparatively simple, sensitive, and affordable [77].

Riley et al. used 3D EEMs to monitor the DOM changes during PW treatment. They used parallel factor analysis (PARAFAC) to decompose the 3D EEMs into chemically independent groups of chromophore components. Their semi-quantified results were validated by several quantitative analytical techniques, including LC-OCD, LC-HRMS, and GC-MS [42]. Wang et al. also used 3D EEMs with fluorescence regional integration (FRI) to characterize and semi-quantify the DOM in FW and PW from Bakken shale, Barnett shale, and DJ basin [78]. The studies proved that 3D fluorescence could be a powerful and inexpensive tool to comprehensively and continuously monitor the DOM in PW, which is essential when the analysis does not target specific organics. It can also facilitate the early detection of system disruptions and guide researchers to choose proper advanced analytical techniques.

7. Inorganic Analysis

Inorganic constituents in PW are often monitored to preserve and protect the equipment in the field. Sodium and chloride are the dominant ions in PW, and high-salinity water corrodes metal pipes and instruments quickly. Additionally, calcium, magnesium, barium, and strontium can form a scale with carbonate and sulfate to decrease the performance of the whole system [79]. Sulfate in PW can also be reduced to hydrogen sulfide, which can corrode infrastructure and is a safety hazard to workers. Elevated levels of boron, iron, and titanium can cause a series of problems during HF, such as over crosslinking, reduced gel viscosity, and inefficient proppant dispersion [30]. Furthermore, the high concentration of ions, especially sodium and chloride, often obstruct the subsequent analysis, treatment, and reuse of PW.

Conductivity and TDS can provide a relative estimation of ions dissolved in PW, while advanced analytical methods are required to measure the composition of individual ions. To analyze anions in PW, such as chloride, bromide, phosphate, nitrate, and sulfate, ion chromatography (IC) is the preferred analytical technique [5]. EPA has two approved methods for anion determination: method 300.0 and 300.1. Method 300.0 is more suitable for PW because it can be applied to industrial wastewater [75]. Currently, the commercial IC system often uses a conductivity detector. Cantlay et al. expanded the IC performance by using ultraviolet/visible light (UV/VIS) and conductivity detectors in tandem, which increased the selectivity for nitrate, nitrite, and bromide in PW samples [80].

For cation analysis, inductively coupled plasma-optical emission spectroscopy (ICP-OES), inductively coupled plasma-atomic emission spectroscopy (ICP-AES), and inductively coupled plasma-mass spectroscopy (ICP-MS) are the preferred methods. They can simultaneously analyze multiple elements in one sample, more convenient than flame atomic absorption (FAA) or graphite furnace atomic absorption (GFAA), which can only analyze one element at a time. Another advantage for ICP-based techniques is that they require minimal sample preparation, often only need filtration, dilution, and addition of acid because the high temperature of an ICP torch can eliminate most of the interferences in the PW matrix [5]. The EPA approved standard methods for ICP-OES and ICP-MS are EPA 200.7 and EPA 200.8/EPA 6020B, respectively [75]. Generally, ICP-MS can provide a detection limit down to parts per trillion (ppt, or ng/L) level in a sample. Cantlay et al. compared the selectivity and sensitivity of ICP-MS and ICP-OES for cation analysis in the high salt matrix of O&G brine from conventional and unconventional wells. The results demonstrated that ICP-MS is better for multi-element analysis at sub-ppb levels;

however, ICP-OES can provide single-digit ppb for most elements. They also found the spectral interferences with ICP-OES were minimal even with the high sodium content as it emits in the high visible range. However, ICP-MS can be susceptible to polyatomic interferences caused by ions consisting of more than one atom or charge, such as $^{40}Ar^{35}Cl$ and $^{40}Ca^{23}Na$ ions affecting ^{75}As signal and $^{40}Ar^{16}O$ and $^{40}Ca^{16}O$ ions affecting the ^{56}Fe signal. Collision/reaction cells have been applied in quadrupole ICP-MS to remove spectral interferences (matrix and argon-based interferences, EPA Method 6020B). Reaction mode uses specific reaction gases to remove known reactive interferences, while helium collision mode with kinetic energy discrimination is universal; it does not need a specific setup for an analyte or a matrix. [81,82]. Despite ICP-OES being less sensitive, it is less expensive and easier to perform, and most metals in PW are abundant enough to be measured by it. Therefore, the choice between the two methods depends on the targeted metal ions and the sample matrix.

Proper sample preparation, such as filtration using 0.22 µm or 0.45 µm filters and dilution, is always required for inorganic ion analysis to ensure more accurate results. SPE using metal affinity resins has been used to detect trace elements in seawater; it can also be applied to PW analysis [83]. In addition, nitric acid is often used to adjust solution pH to below 2 to stabilize the samples for cation analysis. Series of dilution is often necessary to get the result for each ion [80].

Assessing rare earth elements (REEs) concentrations in geothermal water and PWs has become more critical due to their potential as strategic mineral commodities. While several methods exist for REEs analysis, such as laser-induced breakdown spectroscopy (LIBS) and instrumental neutron activation analysis (INAA), ICP-MS is the predominant method used by researchers [84,85]. Currently, there is no EPA method for REEs analysis. However, the U.S. Geological Survey (USGS) Open File Report 02-223, Chapter K details the measurement of REEs in geological materials [86]. Quillinan et al. [87] applied ICP-MS to analyze REEs in PW in the U.S. Two sophisticated sample preparation methods were developed based on the TDS level to eliminate the impact of high salinity and hydrocarbons and preconcentrate the REEs. One approach is suitable for samples with TDS lower than 4.5 g/L, while another procedure is specified for samples with TDS from 4.5 to 300 g/L. There are other methods for specific ion detection, such as an ion-selective electrode. For example, Xu et al. [88] and Almaraz [47] used an iodide double-junction ion-selective electrode to measure iodide concentration in PW.

8. Microbiological Characterization

The characterization and enumeration of the microbial communities in PW are critical for understanding and limiting the impact on corrosion, fouling, and souring issues, thus protecting well infrastructure, minimizing biocide dosage, and supporting PW reuse. Currently, there are no standards regarding the acceptable levels of bacteria in PW [59]. Different methods, both culture-dependent and culture-independent, can be used to measure bacteria, such as plate counting methods, molecular methods, and matrix-assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI-ToF-MS) [59]. Plate counting methods are the most commonly used in industry, where the samples are placed on different nutrient agar plates and incubated for a certain amount of time. Differential nutrient media containing specific ingredients are used to distinguish selected species or categories of bacteria by visual observation. Several EPA-approved methods are based on plate counting methods, such as the EPA Method 1600 for Enterococci bacteria in water, EPA Method 1603 for *Escherichia coli (E.coli)* bacteria in water [75]. However, the disadvantage of the plate methods is that only 1% of the microorganisms in nature are able to grow in an artificial environment [89]. Moreover, there is still a lack of developed differential nutrient media and cultivation methods to detect many corrosion-causing microorganisms such as sulfate-reducing bacteria, sulfur-oxidizing bacteria, iron-oxidizing/reducing bacteria, sulfate-reducing archaea, methanogenic archaea, and anaerobic fermenting acid-producing bacteria. Thus, molecular methods are developed to aid the identification of unculturable and slow-growing microorganisms.

Molecular methods include nucleic acid-based techniques, and immunological methods have been applied to study the microorganisms in PW. Nucleic acid-based techniques, such as DNA/RNA sequencing and polymerase chain reaction (PCR), use genetic sequences unique to each microorganism for their identification [90]. PCR can usually be used to amplify the small amount of DNA or RNA in a sample for detection. However, it only works for certain groups of microorganisms with designed primers and is not ideal for identifying the whole microbial community. The new next-generation sequencing (NGS) technology has shown significant advantages in analyzing the microbial community for its unprecedented sequencing depth. It has been applied for investigating microbial structure and functions in various complex environments. This metagenome-based approach could offer a more comprehensive view of the genetic complexity of communities, allowing us to better assess the change of microbial taxonomic diversity and metabolic potential within the bacterial community in water samples [91]. 16S metagenomic sequencing method can be used to identify unknown and unculturable microorganisms; thus, it is widely used to analyze bacteria and archaea in PW [78,90]. Immunological methods are based on the ability of antibodies to recognize specific structures (e.g., proteins or polysaccharides) of biological macromolecules. One example of a commonly used immunological method to evaluate bacteria is the enzyme-linked immunosorbent assay (ELISA), which uses enzymatic reactions to detect the antigen-antibody complex [92]. Recent advances have made it a promising tool that can be applied for target analysis of PW [93].

MALDI-ToF-MS is another powerful tool to analyze microorganisms based on their protein profile [30,63,94,95]. The advantages of this technique are that it can provide metabolic states of the detected cells; it has the potential to directly identify each bacterium in simple mixtures without purification procedures [96]. It can also detect bacteria at the species level. Santos et al. investigated bacteria in groundwater near UD and found that 16S rRNA gene sequencing results for *Klebsiella* sp., *Enterobacter* sp., and *Citrobacter* sp. were not conclusive due to the genetic similarity of those bacterial genera under the same family. In this scenario, the MALDI-TOF showed its advantage and identified the same microorganism as *Klebsiella oxytoca* with a 99.9% match score [97]. However, several disadvantages of this technique have limited its wide application. First, it can only identify culturable microorganisms due to its detection limit. Second, the initial capital cost of this technique is high, although its subsequent analyses are more affordable and faster than nucleic acid techniques. In addition, it requires a microorganism database to identify the bacteria by matching the generated peak lists from MS. Thus, it cannot be used to detect the bacteria with protein spectra not listed in the database, and 16S rRNA gene sequencing is often required to assist the identification process in this situation. The commercial MALDI-ToF-MS microorganism database (such as Shimadzu SARAMIS microorganism database) has been developed primarily for clinical applications and often lack some entries for organisms that are more prevalent in the environment [95]. Hildenbrand et al. used MALDI-ToF-MS to identify the microorganisms present in PW for the first time in 2018. However, they still needed to use 16S rRNA sequencing to identify bacteria that were not successfully identified using MALDI-ToF-MS and to confirm the bacterial identification due to the database problem. With the help of 16S rRNA sequencing, researchers keep adding the protein spectra of those previously unidentified organisms into the MS library for subsequent environmental investigations [30].

In summary, the EPA-approved plate counting methods may be suitable for PW, but they only detect minimal types of bacteria. The 16S rRNA gene sequencing and MALDI-ToF MS are the two most important techniques for identifying bacteria in PW. They are complementary techniques: the 16S rRNA gene sequencing is more reliable in the taxonomic organization, while the MALDI-TOF has better resolving power at the species level [96]. Currently, most of the published literature used 16S rRNA alone or MALDI-ToF MS with 16S rRNA to identify microorganisms in PW or groundwater related to UD.

9. Analysis of Naturally Occurring Radioactive Material (NORM)

Produced water usually contains naturally occurring radioactive material (NORM) because organic-rich shale deposits have inherently higher radioactivity than typical rock formations. Their existence associated with health concern was first realized in the 1980s when unacceptable radiation levels were detected by scrap metal dealers [98,99]. Studies indicate that the concentration of NORM in PW increases with salt content, and the abundant chloride especially enhances the solubility of NORM [100,101]. The commonly found NORM in PW includes uranium (U), thorium (Th), thallium (Tl), and radium (Ra). ^{226}Ra (half-lives of 1500 years) and ^{228}Ra (half-lives of 5.75 years) are the most abundant, which come from the decay of ^{238}U (half-lives of 4.5 billion years) and ^{232}Th (half-lives of 14.5 billion years) [12,102]. The content of ^{226}Ra in the concentrated brine trapped in the Marcellus shale can exceed 10,000 pCi/L, while the standard for drinking water (^{226}Ra and ^{228}Ra) is 5 pCi/L [101]. Radium can accumulate on oilfield equipment through coprecipitation with scale deposits, and the most likely host of radium in the subsurface formation is mineral barite ($BaSO_4$) [98,103]. One study in North Dakota found the NORM in different waste streams from O&G activities, while the scale in equipment had the highest abundance [104]. Another attribute of the PW matrix is the occurrence of sulfate-reducing bacteria, as discussed in Section 8, which increases the solubility of $BaSO_4$, leading to the release of previously encapsulated radium [103].

Evaluating the levels of NORM is critical to protect the person handling PW and the environment. There are three major types of radiation-alpha, beta, and gamma-and the detection methods for NORM usually are based on measuring these types of radiation. For example, ^{226}Ra primarily emits alpha particles, which can be measured directly. ^{228}Ra can be determined by measuring the decay product ^{228}Ac [105]. The EPA has the standard method 903.0 to measure the total soluble alpha-emitting radioisotopes of radium in drinking water. Currently, the EPA-approved methods (900.0, 903.0, 903.1, and 901.1) can only be applied to drinking water, not other water-based samples [75]. One study has proved the EPA Method 903.0 to be inaccurate when applied to PW because of the remarkably high ionic strength and TDS [106,107].

High-purity germanium gamma spectrometer (HPGe-GS) has been proven to be a reliable approach to measure Ra activity in PW. However, this method is limited by the available sample size, long preparation time (21 days to allow ^{228}Ra to reach radioactive secular equilibrium), detector efficiency, and available counting time (6–48 h) [106]. ICP-MS is an effective method for detecting radioactive isotopes compared to the traditional method [108]. Zhang et al. used SPE combined with ICP-MS to analyze ^{226}Ra in PW and compared the result with those obtained from gamma spectroscopy. The study indicated that ICP-MS is a rapid and powerful tool to detect ^{226}Ra with recoveries near 100% from PW samples [105]. Thus, recent studies usually combine HPGe-GS with ICP-MS for PW radioactivity analysis. Rosenblum et al. used HPGe-GS for radionuclide analysis (^{226}Ra, ^{210}Pb, ^{210}Po, ^{234}U, and ^{238}U). The counting time was set to 60,000s for all PW samples unfiltered and acidified with nitric acid (except ^{238}U sample). ICP-MS measured the activities of 238U. Ratios of ^{238}U radioactivity concentrations to ^{234}U radioactivity concentrations were determined by alpha spectrometry using the Eichrom method (ACW02) and uranium-232 as a tracer [28].

Fan et al. [109] compared the HPGe-GS and ICP-MS performance by analyzing ^{226}Ra in PW samples from Antrim and Utica-Collingwood shale (MI) and Marcellus shale (PA). They found that despite the rapid analysis (same day results), ICP-MS measurement of ^{226}Ra did not perform well at high salinity (e.g., 150–230 g/L chloride)/low Ra activity (e.g., <1000 pCi/L) compared to HPGe-GS. However, ICP-MS results had good agreement with HPGe-GS at a lower salinity and higher Ra activity. Moreover, the detection limit of ICP-MS (~1250 pCi/L) is ten times higher than that of the HPGe-GS method. However, because many PW samples fall within the operational salinity range and Ra activity (or with proper sample treatment, dilution, and SPE) of the ICP-MS method, ICP-MS will still be the more efficient method for estimating Ra in PW samples.

10. Summary, Knowledge Gap, and Research Needs

Managing PW, including onsite reuse and beneficial uses outside the oil and gas field after treatment, remains a significant challenge for producers, operators, and regulators due to a lack of complete understanding of the constituents in PW. Risk assessment for PW reuse also heavily relies on knowing the constituents with their concentrations in the water matrix. Therefore, it is crucial to develop a comprehensive understanding of the analytical methods to characterize the known and unknown compounds and to standardize the existing methods suitable for PW analysis with modification and verification.

A multi-tiered analytical approach is proposed to characterize PW quality at all stages and levels of the water cycle from monitoring process performance, modeling potential technology applicability, and evaluating treatment efficiency to assessing environmental and public health risks associated with fit-for-purpose applications and disposal of waste streams. There is a need for developing robust, quick, efficient, accurate, sensitive, and cost-effective analytical methods for characterization of important produced water constituents for screening, treatment process monitoring, pre- and post-treatment quality assurance, application monitoring, and regulatory compliance. Both rapid testing techniques to support process quality assurance (QA) and advanced analytical methods for target and nontarget chemical analysis are required to manage, dispose of, treat, and reuse PW properly. Reliable, accurate, and robust in-line sensors should be developed as Tier 1 analysis of complex produced water chemistry under a harsh environment. Advanced software tools, machine learning, and connected digital systems are also needed to enable data processing, failure analysis, predicting performance, and operation control. Tier 1 analytical results will trigger and guide Tiers 2 to 4 characterization analysis. Tiers 2 to 4 focus on detailed characterization, routine monitoring, and Tier 1 data verification.

Current EPA standard methods are developed for drinking water, municipal, and industrial wastewater. Their application to characterizing PW samples needs to be evaluated. The EPA methods for bulk measurements can be suitable for PW samples with minimal revision; the methods for inorganic measurements can be suitable for PW samples if the interference of high TDS is addressed (e.g., series dilution). For NORM and biological analysis, the current EPA methods will not be sufficient. Sample preparation and long measuring time are two major problems that need to be addressed. Further, the prevalent challenge for PW is the analysis of organics, especially for non-volatile compounds and unknown chemicals. GC/MS-based EPA methods with modification, such as intense sample preparation, may be suitable to analyze VOCs and SVOCs in PW, especially with the help of GC-(EI)-MS database and good reproducibility of GC-(EI)-MS. For non-volatile analysis, LC-HRMS/MS will play a crucial role in identifying unknown compounds in PW from additives or transformation products and quantifying the known compounds. Multi-step sample preparation would tremendously assist the analysis of non-VOCs. However, because of the extremely complex matrix of PW and the limitation of the current LC-HRMS/MS technique, it is still difficult to monitor all the organics in the PW and measure their concentrations to assess the potential risks. Complete current MS database or construct a designated database for PW would significantly assist non-VOCs identification and quantification.

The review results are illustrated in Figure 4, summarizing the proposed approaches for analyzing chemical constituents in PW, including sampling and preservation, sample preparation and pretreatment, and analytical techniques. Table 4 summarizes the standard analytical methods, methods used in commercial labs, and advanced characterization research methods for targeted and unknown compounds. Currently, there is no EPA-approved method for PW samples. EPA-approved methods target known chemicals. The unknown composites in PW require nontargeted analysis, especially for organic compounds. More research is needed to develop these proposed approaches to meet the EPA requirements for regulatory purposes.

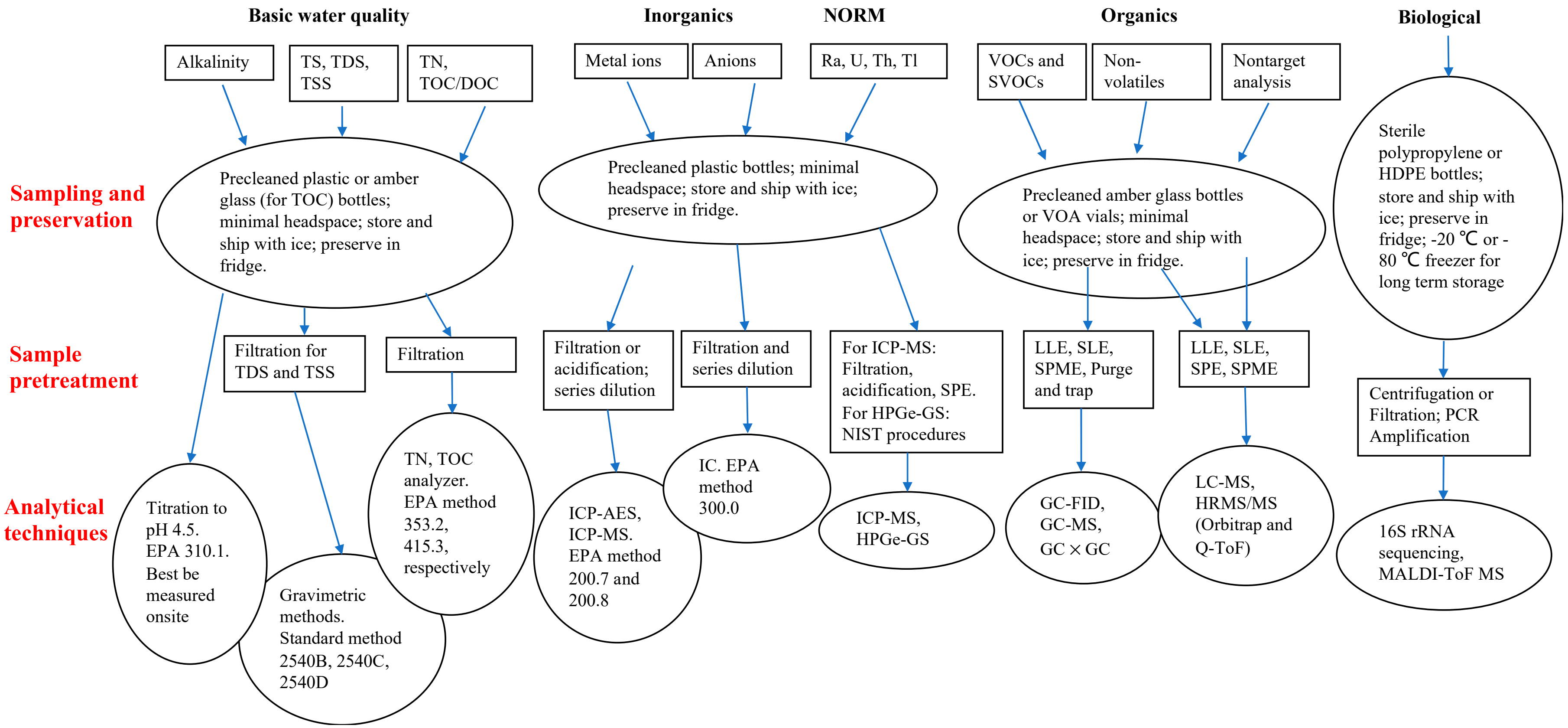

Figure 4. Proposed approaches for analysis of chemical constituents in flowback and produced water.

Table 4. Summary of standard analytical methods and methods used in commercial labs, and advanced characterization research methods for targeted and unknown compounds.

Analytes	EPA-Approved Methods (Water Matrix)	Suitable for PW Analysis?
Basic water quality		
Alkalinity	EPA Method 310.1 and 310.2 (Drinking, surface, and saline waters; domestic and industrial wastes)	EPA Method 310.1 is suitable for PW.
TS	Standard method 2540B (Drinking, surface, and saline waters; domestic and industrial wastewaters)	Yes, range up to 20,000 mg/L
TDS	Standard method 2540C (Drinking, surface, and saline waters; domestic and industrial wastewaters)	Yes, range up to 20,000 mg/L
TSS	Standard method 2540D (Drinking, surface, and saline waters; domestic and industrial wastewaters)	Yes, range up to 20,000 mg/L
TN	EPA Method 353.2: Inorganic nitrite and nitrate; EPA method 351.2 and 351.4: organic nitrogen and ammonia (drinking, surface, and saline waters; domestic and industrial wastes)	Yes, with proper sample preparation
TOC/DOC	EPA Method 415.3 or Standard methods 5310C (Source waters and drinking water)	Yes, with proper sample preparation
pH	EPA 150.1 (Drinking, surface, and saline waters; domestic and industrial wastes and acid rain)	Yes, with proper sample preparation
Inorganic		
Metal ions	EPA 200.7: ICP-AES, EPA 200.8/6020 B: ICP-MS (Drinking, surface, and groundwater; wastewaters, sludges, and solid samples)	EPA methods can be used for PW with a series of dilutions to eliminate the impact of high Na^+ concentration. ICP-AES and ICP-OES are reliable approaches.
Anions	EPA 300.0 (drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids); EPA 300.1 (reagent water, surface water, groundwater, finished drinking water)	EPA 300.0 is suitable for PW with a series of dilutions to eliminate the impact of high Cl^- concentration. IC is a reliable approach.
Organic		
Non-Pesticide (120 parameters)	EPA 551, 601–625, 632, 1613B, etc. (drinking, ambient water, wastewater, sediment)	EPA methods based on GC may be suitable to analyze VOCs and SVOCs in PW with proper sample pretreatment, such as purge and trap, LLE, SPE, or SPME. However, the number of compounds is limited.
Pesticide (70 parameters)	EPA 553, 605, 610, etc. (drinking, ambient water, wastewater, sediment)	With proper sample pretreatment, such as LLE, SPE, or SPME, EPA methods based on LC may be suitable to analyze non-volatiles compounds in FPW. However, LC-HRMS/MS (Orbitrap and Q-ToF) would be more reliable approaches.
Nontarget analysis	No methods.	Nontarget analysis using HRMS/MS (confidence Levels 1 and 2) will be required to identify the unknown compounds in PW.
Biological Bacterial	EPA 1600, 1603, 1622, 1680, etc. (Wastewater and Sewage sludge, ambient water)	EPA methods detect limited types of bacteria. 16S rRNA sequencing and MALDI-ToF MS are reliable approaches for FPW.
NORM Ra, U, Th, Tl	EPA 900.0, 901.1, 903.0, 903.1. (Drinking water)	EPA methods cannot be used for PW. HPGe-GS (lower MDL and more accurate) and ICP-MS (more efficient) are reliable approaches.

Supplementary Materials: The following are available online at https://www.mdpi.com/2073-4441/13/2/183/s1, Figure S1: Temporal variation of PW qualities in Marcellus shale, PA, two well sites; and Niobrara formation, CO, two well sites. Table S1:Comparison of general physicochemical parameters of PW in primary UD plays in minimum-maximum/mean values; Table S2: Analyte containers, preservation, and holding times; Table S3: SPE cartridges used for organic extraction; Table S4: Summary of the recent studies analyzing organic compounds in PW.

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Nomenclature

AEOs	Alkyl ethoxylates
APCI	Atmospheric pressure chemical ionization
ASE	Accelerated solvent extraction
AST	Above surface storage tank
BOD	Biochemical oxygen demand
BSFTA	N, O-bis(trimethylsilyl)trifluoroacetamide
BTEX	Benzene, toluene, ethylbenzene, and xylenes
CI	Chemical ionization
COD	Chemical oxygen demand
DCM	Dichloromethane
DOC	Dissolved organic carbon
DOM	Dissolved organic matter
EI	Electron ionization
EPA	Environmental Protection Agency
ESI	Electrospray ionization
FEEMs	Fluorescence excitation-emission matrix
FID	Flame ionization detector
FPSE	Fabric phase sorptive extraction
FT-ICR-MS	Fourier-transform ion cyclotron resonance mass spectrometry
FW	Flowback water
GC	Gas chromatography
GC-FID	Gas chromatography coupled with flame ionization detector
GC-MS	Gas chromatography-mass spectrometry
GC-TCD	Gas chromatography coupled with thermal conductivity detector
HDPE	High-density polyethylene
HF	Hydraulic fracturing
HPGe-GS	High-purity germanium gamma spectrometer
HPLC	High performance liquid chromatography
HPLC-MS	High performance liquid chromatography -mass spectroscopy
HPLC-UV	High performance liquid chromatography coupled with ultraviolet diode array detector
HRMS	High-resolution mass spectrometry
HSGC	Headspace gas chromatography
IC	Ion chromatography
ICP-AES	Inductively coupled plasma-atomic emission spectroscopy
ICP-MS	Inductively coupled plasma-mass spectroscopy
ICP-OES	Inductively coupled plasma-optical emission spectroscopy
LC	Liquid chromatography
LC-MS	liquid chromatography-mass spectroscopy
LC-OCD	liquid chromatography coupled with organic carbon detection
LC-UV	liquid chromatography coupled with UV-diode array detector
LEAF	Leaching environmental assessment framework
LLE	Liquid-liquid extraction
MALDI	Matrix-assisted laser desorption/ionization
MDL	Minimum detection limit
MS	Mass spectroscopy
MS/MS	Tandem mass spectrometry
NORM	Naturally occurring radioactive material
O&G	Oil and gas
ORP	Oxidation-reduction potential
PAHs	Polycyclic aromatic hydrocarbons
PEGs	Polyethylene glycols
PTFE	Polytetrafluoroethylene
PW	Produced water
Q	Quadrupole mass analyzer
REEs	Rare earth elements
SEM/EDX	Scanning electron microscopy/energy-dispersive X-ray spectroscopy
SI	Supporting Information
SLE	Solid-liquid extraction
SPE	Solid-phase extraction
SPLP	Synthetic precipitation leaching procedure
SPME	Solid-phase microextraction
SVOCs	Semi-volatile organic compounds
SWD	Salt water disposal
TCLP	Toxicity characteristic leaching procedure;
TDS	Total dissolved solids
TN	Total nitrogen
TOC	Total organic carbon
ToF	Time of flight mass analyzer
TPH	Total petroleum hydrocarbons
TSS	Total suspended solids
UD	Unconventional oil and gas development
UIC	Underground injection control
VOA	Volatile organic analysis
VOCs	Volatile organic compounds
XRD	X-ray diffraction

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EXHIBIT WG-6

Hydraulic Fracturing Fluid Product Component Information Disclosure

Job Start Date:	4/7/2015
Job End Date:	4/7/2015
State:	New Mexico
County:	Eddy
API Number:	30-015-42770-00-00
Operator Name:	Devon Energy Production Company L. P.
Well Name and Number:	Cotton Draw Unit 99
Longitude:	-103.72987400
Latitude:	32.18003000
Datum:	NAD27
Federal/Tribal Well:	NO
True Vertical Depth:	10,529
Total Base Water Volume (gal):	182,980
Total Base Non Water Volume:	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Fresh Water	Operator	Base Fluid					
			Fresh Water	7732-18-5	100.00000	87.54031	Density = 8.340
SAND - PREMIUM WHITE	Halliburton	Proppant					
			Crystalline silica, quartz	14808-60-7	100.00000	8.57363	
SSA-2	Halliburton	Proppant					
			Crystalline silica, quartz	14808-60-7	100.00000	2.52287	
FE-1	Halliburton	Additive					
			Acetic acid	64-19-7	100.00000	0.99010	
WG-36 GELLING AGENT	Halliburton	Gelling Agent					
			Guar gum	9000-30-0	100.00000	0.07182	
OILPERM FM-7	Halliburton	Surfactant					
			Poly(oxy-1,2-ethanediyl), .alpha.-isodecyl-.omega.hydroxy-	61827-42-7	10.00000	0.00726	
			Isopropanol	67-63-0	10.00000	0.00726	
			Terpenes and Terpenoids, sweet orange-oil	68647-72-3	10.00000	0.00726	Density = 8.217
			Fatty acids, coco, reaction products with ethanolamine, ethoxylated	61791-08-0	10.00000	0.00726	

			Terpene hydrocarbon by-products	68956-56-9	5.00000	0.00363	
			Glycerine	56-81-5	5.00000	0.00363	
Cla-Web(TM)	Halliburton	Additive					
			Ammonium salt	Confidential	60.00000	0.03247	Denise Tuck, Halliburton, 3000 N. Sam Houston Pkwy E., Houston, TX 77032 (281) 871-6226
CL-22 UC	Halliburton	Crosslinker					
			Potassium formate	590-29-4	60.00000	0.02305	
FR-66	Halliburton	Friction Reducer					
			Hydrotreated light petroleum distillate	64742-47-8	30.00000	0.02219	
HAI-OS ACID INHIBITOR	Halliburton	Corrosion Inhibitor					
			Methanol	67-56-1	60.00000	0.01071	
			Propargyl alcohol	107-19-7	10.00000	0.00179	
CL-31 CROSSLINKER	Halliburton	Crosslinker					
			Potassium metaborate	13709-94-9	60.00000	0.00901	
			Potassium hydroxide	1310-58-3	5.00000	0.00075	
OptiKleen-WF(TM)	Halliburton	Concentrate					
			Sodium perborate tetrahydrate	10486-00-7	100.00000	0.00723	
MO-67	Halliburton	pH Control Additive					
			Sodium hydroxide	1310-73-2	30.00000	0.00439	
OPTIFLO-III DELAYED RELEASE BREAKER	Halliburton	Breaker					
			Ammonium persulfate	7727-54-0	100.00000	0.00304	
			Crystalline silica, quartz	14808-60-7	30.00000	0.00091	
BE-3S BACTERICIDE	Halliburton	Biocide					
			2,2 Dibromo-3-nitrilopropionamide	10222-01-2	100.00000	0.00207	
			2-Monobromo-3-nitrilopropionamide	1113-55-9	5.00000	0.00010	
BE-6 MICROBIOCIDE	Halliburton	Biocide					
			2-Bromo-2-nitro-1,3-propanediol	52-51-7	100.00000	0.00207	
SP BREAKER	Halliburton	Breaker					
			Sodium persulfate	7775-27-1	100.00000	0.00029	
Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.							
		Other Ingredient(s)					
			Water	7732-18-5		0.11200	
			Miscellaneous	NA	0.50000	0.03629	OilPerm FM-7
			Borate salts	Confidential		0.02305	
			Polyacrylamide copolymer	Confidential		0.02219	
			Sodium chloride	7647-14-5		0.00693	
			Alcohols, C14-C15, ethoxylated	68951-67-7		0.00536	

			Reaction product of acetophenone, formaldehyde, thiourea and oleic acid in dimethyl formamide	68527-49-1		0.00536	
			Fatty acids, tall oil	Confidential		0.00536	
			Amide	Confidential		0.00370	
			Ammonium chloride	12125-02-9		0.00370	
			Alcohols ethoxylated	Confidential		0.00370	
			Bentonite, benzyl(hydrogenated tallow alkyl) dimethylammonium stearate complex	121888-68-4		0.00359	
			Quaternary amine	Confidential		0.00271	
			Cured acrylic resin	Confidential		0.00091	
			Olefins	Confidential		0.00089	
			Olefins	Confidential		0.00089	
			Sorbitan monooleate polyoxyethylene derivative	9005-65-6		0.00074	
			Sorbitan, mono-9-octadecenoate, (Z)	1338-43-8		0.00074	
			Surfactant mixture	Confidential		0.00072	
			Silica gel	112926-00-8		0.00072	
			Surfactant mixture	Confidential		0.00072	
			Quaternary amine	Confidential		0.00054	
			Acrylate polymer	Confidential		0.00038	
			Sodium carboxymethyl cellulose	9004-32-4		0.00038	
			Sodium glycollate	2836-32-0		0.00038	
			Olefins	Confidential		0.00018	
			Olefins	Confidential		0.00018	
			Crystalline silica, quartz	14808-60-7		0.00011	
			Quaternary amine	Confidential		0.00005	
			Amine salts	Confidential		0.00005	
			Amine salts	Confidential		0.00005	
			Sodium sulfate	7757-82-6		0.00000	

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Hydraulic Fracturing Fluid Product Component Information Disclosure

Job Start Date:	5/6/2015
Job End Date:	5/7/2015
State:	New Mexico
County:	Eddy
API Number:	30-015-42770-00-00
Operator Name:	Devon Energy Production Company L. P.
Well Name and Number:	Cotton Draw Unit 99
Longitude:	-103.72987400
Latitude:	32.18003000
Datum:	NAD27
Federal/Tribal Well:	NO
True Vertical Depth:	10,529
Total Base Water Volume (gal):	193,381
Total Base Non Water Volume:	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Fresh Water	Operator	Base Fluid					
			Fresh Water	7732-18-5	100.00000	88.34860	Density = 8.340
BE-6, BE-9, CL-22 UC, CL-31 Crosslinker, Cla-Web(TM), FR-66, MO-67, Oilperm FM-7, OptiFlo-III, OptiKleen-WF(TM), Sand-Common White-100 Mesh, SSA-2, Sand-Premium White-40/70, SP Breaker, WG-36 Gelling Agent	Halliburton	Additive, Biocide, Breaker, Concentrate, Crosslinker, Friction Reducer, Gelling Agent, Microbiocide, pH Control Additive, Proppant					
			Crystalline silica, quartz	14808-60-7	100.00000	10.43094	
			Water	7732-18-5	100.00000	0.13205	
			Guar gum	9000-30-0	100.00000	0.06135	
			Ammonium salt	Confidential	60.00000	0.02920	
			Polyacrylamide copolymer	Confidential	30.00000	0.01756	
			Hydrotreated light petroleum distillate	64742-47-8	30.00000	0.01756	
			Borate salts	Confidential	60.00000	0.01545	
			Potassium formate	590-29-4	60.00000	0.01545	

			Sodium perborate tetrahydrate	10486-00-7	100.00000	0.00756	
			Potassium metaborate	13709-94-9	60.00000	0.00645	
			Sodium chloride	7647-14-5	5.00000	0.00576	
			Sodium hydroxide	1310-73-2	30.00000	0.00419	
			Tributyl tetradecyl phosphonium chloride	81741-28-8	10.00000	0.00396	
			Bentonite, benzyl(hydrogenated tallow alkyl) dimethylammonium stearate complex	121888-68-4	5.00000	0.00307	
			Quaternary amine	Confidential	5.00000	0.00297	
			Amide	Confidential	5.00000	0.00293	
			Ammonium chloride	12125-02-9	5.00000	0.00293	
			Alcohols ethoxylated	Confidential	5.00000	0.00293	
			Ammonium persulfate	7727-54-0	100.00000	0.00274	
			2-Bromo-2-nitro-1,3-propanediol	52-51-7	100.00000	0.00197	
			Surfactant mixture	Confidential	1.00000	0.00123	
			Cured acrylic resin	Confidential	30.00000	0.00082	
			Silica gel	112926-00-8	1.00000	0.00061	
			Sorbitan, mono-9-octadecenoate, (Z)	1338-43-8	1.00000	0.00059	
			Sorbitan monooleate polyoxyethylene derivative	9005-65-6	1.00000	0.00059	
			Sodium persulfate	7775-27-1	100.00000	0.00055	
			Potassium hydroxide	1310-58-3	5.00000	0.00054	
			Sodium carboxymethyl cellulose	9004-32-4	1.00000	0.00026	
			Acrylate polymer	Confidential	1.00000	0.00026	Denise Tuck, Halliburton, 3000 N. Sam Houston Pkwy E., Houston, TX 77032, 281-871-6226
			Sodium glycollate	2836-32-0	1.00000	0.00026	
			Amine salts	Confidential	0.10000	0.00010	
			Crystalline Silica, Quartz	14808-60-7	0.10000	0.00006	
			Sodium sulfate	7757-82-6	0.10000	0.00000	
HCl < 10%	Halliburton	Base Fluid					
			Hydrochloric Acid	7647-01-0	100.00000	0.94988	

Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

Hydraulic Fracturing Fluid Product Component Information Disclosure

Job Start Date:	7/14/2015
Job End Date:	7/16/2015
State:	New Mexico
County:	Eddy
API Number:	30-015-42770-00-00
Operator Name:	Devon Energy Production Company L. P.
Well Name and Number:	Cotton Draw Unit 99
Longitude:	-103.72990000
Latitude:	32.17997000
Datum:	NAD27
Federal/Tribal Well:	NO
True Vertical Depth:	9,162
Total Base Water Volume (gal):	229,992
Total Base Non Water Volume:	0

Hydraulic Fracturing Fluid Composition:

Trade Name	Supplier	Purpose	Ingredients	Chemical Abstract Service Number (CAS #)	Maximum Ingredient Concentration in Additive (% by mass)**	Maximum Ingredient Concentration in HF Fluid (% by mass)**	Comments
Water	Operator	Carrier					
			Water	7732-18-5	100.00000	95.44868	
Sand, White, 40/70	Baker Hughes	Proppant					
			MSDS and Non-MSDS Ingredients Listed Below	NA		1.57067	
Sand, White, 100 mesh	Baker Hughes	Proppant					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.78978	
HCl, 5.1 - 7.5%	Baker Hughes	Acidizing					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.64700	SmartCare Product
BF-7L	Baker Hughes	Buffer					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.27031	
GW-3D	Baker Hughes	Gelling Agent					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.23104	
Sand, White, 30/50	Baker Hughes	Proppant					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.19670	
XLW-30G, tote	Baker Hughes	Crosslinker					

			MSDS and Non-MSDS Ingredients Listed Below	NA		0.15165	
InFlo 72	Baker Hughes	Surface Tension Reducer					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.13682	
ClayCare, tote	Baker Hughes	Clay Control					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.13010	SmartCare Product
MaxPerm-20A, 265 gallon tote	Baker Hughes	Friction Reducer					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.04414	SmartCare Product
XLW-4	Baker Hughes	Crosslinker					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.04024	
Alpha 125	Baker Hughes	Biocide					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.03358	SmartCare Product
Parasorb 5000, bag	Baker Hughes	Paraffin Inhibitor					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.00998	SmartCare Product
GBW-5	Baker Hughes	Breaker					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.00749	SmartCare Product
Ferrotrol 280L	Baker Hughes	Iron Control					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.00321	
High Perm CRB	Baker Hughes	Breaker					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.00250	SmartCare Product
CI-27	Baker Hughes	Corrosion Inhibitor					
			MSDS and Non-MSDS Ingredients Listed Below	NA		0.00148	
Ingredients shown above are subject to 29 CFR 1910.1200(i) and appear on Material Safety Data Sheets (MSDS). Ingredients shown below are Non-MSDS.							
Ingredients in Additive (s) (MSDS and non-MSDS)	Baker Hughes	See Trade Name(s) List					
			Crystalline Silica (Quartz)	14808-60-7	100.00000	2.54964	
			Water	7732-18-5	99.00000	1.10259	
			Guar Gum	9000-30-0	100.00000	0.23036	
			Potassium Carbonate	584-08-7	60.00000	0.16170	
			Choline Chloride	67-48-1	75.00000	0.09728	
			Ulexite Mineral	1319-33-1	40.00000	0.06048	
			Hydrochloric Acid	7647-01-0	7.50000	0.04838	
			Potassium Formate	590-29-4	30.00000	0.04536	
			Polyethers	68815-65-6	30.00000	0.04092	
			Isopropanol	67-63-0	30.00000	0.04092	
			1-Propanesulfonic acid, 2-methyl-2-[(1-oxo-2-propen-1-yl) amino]-, polymer with 2-propenamide, sodium salt	83446-68-8	60.00000	0.02640	
			Organic Polyol	112-27-6	15.00000	0.02046	

			Nonionic Alkoxylate	70559-25-0	10.00000	0.01364	
			Hydrotreated Light Distillate	64742-47-8	30.00000	0.01320	
			Sodium Tetraborate	1303-96-4	30.00000	0.01204	
			Glutaraldehyde	111-30-8	30.00000	0.01004	
			Glycerin	8043-29-6	25.00000	0.01003	
			Ammonium Persulphate	7727-54-0	100.00000	0.00995	
			Diatomaceous Earth, Calcined (kieselguhr)	91053-39-3	70.00000	0.00697	
			Orange Terpene	68647-72-3	5.00000	0.00682	
			Surfactant	68131-39-5	1.50000	0.00477	
			Clay	12174-11-7	3.00000	0.00454	
			2-Mercaptoethanol	60-24-2	100.00000	0.00320	
			Cellulose	9004-32-4	2.00000	0.00302	
			Potassium Hydroxide	1310-58-3	1.00000	0.00270	
			Sodium Chloride	7647-14-5	5.00000	0.00223	
			Sorbitan, mono-(9Z)-9-octadecenoate	1338-43-8	5.00000	0.00220	
			Polyoxyethylene sorbitan monooleate	9005-65-6	5.00000	0.00220	
			Oxyalkylated alcohol	78330-21-9	5.00000	0.00220	
			Sodium bisulfate	7681-38-1	1.00000	0.00151	
			Sodium formaldehyde bisulfate	870-72-4	1.00000	0.00151	
			Acrylamide-acrylic acid copolymer, sulfomethylated,	EPA Acct 108431	1.00000	0.00151	
			Crystalline Silica Quartz	14808-60-7	1.00000	0.00151	
			Sodium glycolate	2836-32-0	1.00000	0.00151	
			Sodium chloride	7647-14-5	1.00000	0.00151	
			Acetic acid ethenyl ester, polymer with ethene	24937-78-8	15.00000	0.00149	
			Mineral Oil	8042-47-5	15.00000	0.00149	
			Diatomaceous Earth, Natural (kieselguhr)	61790-53-2	10.00000	0.00100	
			Methanol	67-56-1	60.00000	0.00097	
			Vinylidene Chloride-Methyl Acrylate Polymer	25038-72-6	25.00000	0.00062	
			Thiourea Polymer	68527-49-1	30.00000	0.00044	
			Fatty Acids	61790-12-3	30.00000	0.00044	
			Polyoxyalkylenes	68951-67-7	30.00000	0.00044	
			Glycerol	56-81-5	0.50000	0.00021	
			Beta-Mannanases	37288-54-3	0.45000	0.00019	
			Ammonium Hydroxide	1336-21-6	5.00000	0.00016	
			Cupric Chloride	7447-39-4	5.00000	0.00016	
			Propargyl Alcohol	107-19-7	10.00000	0.00015	
			Crystalline Silica (Cristobalite)	14464-46-1	1.00000	0.00010	
			Olefin	64743-02-8	5.00000	0.00007	
			Hydrated Magnesium Silicate	14807-96-6	1.00000	0.00002	
			Poly (Tetrafluoroethylene)	9002-84-0	0.50000	0.00001	
			Magnesium chloride	7786-30-3	0.00100	0.00000	

			5-Chloro-2-methyl-3(2H)-Isothiazolone	26172-55-4	0.00100	0.00000	
			2-methyl-4-isothiazolin-3-one	2682-20-4	0.00100	0.00000	
			Magnesium nitrate	10377-60-3	0.00100	0.00000	
			Formaldehyde	50-00-0	0.05000	0.00000	

* Total Water Volume sources may include fresh water, produced water, and/or recycled water
** Information is based on the maximum potential for concentration and thus the total may be over 100%

Note: For Field Development Products (products that begin with FDP), MSDS level only information has been provided.
Ingredient information for chemicals subject to 29 CFR 1910.1200(i) and Appendix D are obtained from suppliers Material Safety Data Sheets (MSDS)

EXHIBIT WG-7

PSR
PHYSICIANS FOR SOCIAL RESPONSIBILITY
1985 NOBEL PEACE PRIZE
U.S. AFFILIATE OF INTERNATIONAL PHYSICIANS FOR THE PREVENTION OF NUCLEAR WAR

FRACKING WITH "FOREVER CHEMICALS" IN NEW MEXICO

Evidence Shows Oil and Gas Companies Have Used PFAS in New Mexico Wells; Water Risks Especially High for Groundwater-Dependent State

By Dusty Horwitt, J.D.
and Barbara Gottlieb

Data Analysis by Gary Allison

April 12, 2023

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Cover photo: Williams Field Services Natural Gas Processing Facility, Bloomfield, New Mexico, Oct. 2012. Photo credit: Jane Pargiter, EcoFlight.

Maps by Matt Kelso, FracTracker Alliance

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EXECUTIVE SUMMARY

Previously unpublicized information unearthed by Physicians for Social Responsibility (PSR) shows that since at least 2013, oil and gas companies used in New Mexico's oil and gas wells a class of extremely toxic and persistent chemicals known as PFAS. However, gaps in New Mexico's disclosure rules prevent the public from knowing how widely PFAS – or other toxic chemicals – have been used. These findings raise concerns that New Mexicans may unknowingly be exposed to highly hazardous substances that are toxic in minuscule amounts.

PFAS are a class of chemicals known for their toxicity at extraordinarily low levels, their multiple negative health effects including cancer, and their persistence in the environment, leading to their nickname, "forever chemicals." Using these chemicals may be particularly risky in a state where 80 percent of the population depends on groundwater for drinking water. Oil and gas production and waste disposal operations can contaminate groundwater with toxic chemicals including PFAS – and, once contaminated, groundwater is particularly difficult to clean up.

The present report is based on data publicly disclosed by the oil and gas industry regarding the use of chemicals in the stage of oil and gas operations known as hydraulic fracturing, or fracking. We found that between 2013 and 2022, oil and gas companies injected more than 200 oil and gas wells in six counties, in both the Permian and San Juan Basins, with the PFAS known as PTFE (marketed as Teflon). Oil and gas companies also injected wells in Lea County in the Permian Basin with the PFAS called fluoroalkyl alcohol substituted polyethylene glycol.

However, the number of cases of PFAS use we have been able to definitively identify in New Mexico oil and gas extraction may significantly underrepresent the reality. That is in large part because New Mexico law allows oil and gas companies to withhold fracking chemical identities from the public and regulators by claiming them as "trade secrets."

Between 2013 and 2022, oil and gas companies disclosed their use of fracking chemicals in 9,066 oil and gas wells. Of those wells, the companies injected more than 8,200 (over 90 percent) with at least one trade secret chemical per well. Trade secret chemicals used over this period totaled more than 240 million pounds. Information about these chemicals was limited, but scientific experts told PSR that chemicals injected into two dozen wells in the Permian Basin were PFAS, may be PFAS, or are precursor chemicals that could degrade into PFAS. Oil and gas companies injected more than 3,600 of the 8,200 wells with surfactants that could be fluorosurfactants, a class of chemical that include multiple PFAS. Should only a fraction of the unidentified chemicals used in New Mexico's oil and gas wells be PFAS, they could pose a significant threat. (An interactive map showing the locations of wells injected with PFAS and trade secret chemicals is https://psr.org/new-mexico-pfas-map/ Users can zoom in to identify wells near them.)

By shielding from public view the chemicals injected into oil and gas wells, weak disclosure rules raise the potential that New Mexicans may be directly exposed, or their groundwater and well water may be exposed, to PFAS (and other toxic chemicals) from hundreds or even thousands of oil and gas wells and waste disposal sites.

In light of these findings, PSR recommends the following:

- **Halt PFAS use in oil and gas extraction.** New Mexico should follow the lead of Colorado, a major oil- and gas-producing state that in June 2022 passed legislation banning the use of PFAS in oil and gas wells. Furthermore, New Mexico and the U.S. Environmental Protection Agency (EPA) should prohibit PFAS from being used, manufactured, or imported for oil and gas extraction. Many PFAS are replaceable with less-persistent and less-toxic alternatives.
- **Expand public disclosure.** New Mexico should greatly expand its requirements for public disclosure of oil and gas chemicals. TThe state could again follow the example offered by Colorado by requiring disclosure of all individual chemicals used in oil and gas wells, without

exceptions for trade secrets, while still protecting chemical product formulas. New Mexico should also require disclosure on the part of chemical manufacturers and require chemical disclosure prior to permitting, as have California, West Virginia, and Wyoming.

- **Increase testing and tracking.** New Mexico and/or the U.S. EPA should determine where PFAS have been used in oil and gas operations in the state and where related wastes have been deposited. They should test nearby residents, water, soil, flora, and fauna for PFAS, both for the particular type(s) of PFAS used and for organic fluorine to detect the presence of other PFAS. and/or their breakdown products. Testing equipment should be used that is sensitive enough to detect PFAS at a level of single-digit parts per trillion or lower.

- **Require funding and cleanup**. Oil and gas and chemical firms should be required to fund environmental testing for PFAS in their areas of operation, and should PFAS be found, be required to fund cleanup. If water cleanup is impossible, companies responsible for the use of PFAS should pay for alternative sources of water for household and agricultural uses, as needed.

- **Remove New Mexico's oil and gas hazardous waste exemption.** New Mexico exempts oil and gas industry wastes from state hazardous waste rules. New Mexico should follow New York's lead and remove its state-level hazardous waste exemption for the oil and gas industry.

- **Reform New Mexico's regulations for oil and gas production wells and underground injection disposal wells.** The state should prohibit production wells and underground wastewater disposal wells close to underground sources of drinking water, homes, health care facilities and schools, require groundwater monitoring for contaminants near the wells, and for disposal wells, require full public disclosure of chemicals in the wastewater.

- **Transition to renewable energy and better regulation.** Given the use of highly toxic chemicals in oil and gas extraction, including but not limited to PFAS, as well as climate impacts of oil and gas extraction and use, New Mexico should transition away from oil and gas production and move toward renewable energy and efficiency while providing economic support for displaced oil and gas workers. As long as drilling and fracking continue, the state should better regulate these practices so that New Mexicans are not exposed to toxic substances and should empower local governments also to regulate the industry. When doubt exists as to the existence or danger of contamination, the rule of thumb should be, "First, do no harm."

Ruins at Chaco Culture National Historic Park, near Nageezi, New Mexico, Sept. 2009. Photo Credit: SkybirdForever, https://commons.wikimedia.org/wiki/File:Chaco_Canyon_-_Pueblo_Bonito_kiva_and_ruins.JPG.

CH. 1

PFAS: A MANMADE THREAT TO HEALTH AND THE ENVIRONMENT USED IN NEW MEXICO’S OIL AND GAS WELLS

a. PFAS Used in New Mexico Wells

Physicians for Social Responsibility (PSR) has identified evidence from publicly reported oil and gas industry records that a highly dangerous class of chemicals, known as per- and polyfluoroalkyl substances (PFAS), has been used in New Mexico’s oil and gas* wells for hydraulic fracturing (“fracking”). PFAS are known for their toxicity at extremely low levels,[1] their multiple negative health effects including cancer,[2] and their persistence in the environment, which has endowed them with their nickname, “forever chemicals.”[3] Fracking is the stage of oil and gas operations that typically involves high-pressure injections into oil and gas wells of up to tens of millions of gallons of water, sand, and chemicals to fracture rock formations and free up trapped oil and gas.[4]** It is possible that PFAS have been used in additional stages and methods of oil and gas production in New Mexico.

The likely use of PFAS in oil and gas production in New Mexico was first exposed in 2021, initially in a report by PSR[5] and subsequently by Public Employees for Environmental Responsibility.[6] Based on fracking chemical disclosures made to the state and to the nongovernmental organization FracFocus, PSR is now able to identify New Mexico oil and gas wells definitively known to have been injected with PFAS between 2013 and 2022. They include 227 wells in six counties that were injected with PTFE, also known as Teflon and identified by the U.S. Environmental Protection Agency (EPA) as a PFAS.[7] Another 34 wells in Lea county were injected with fluoroalkyl alcohol substituted polyethylene glycol, also identified as a PFAS by EPA.[8] (See chapter 2 for details on these chemicals.) In reaching definitive conclusions about these chemicals, PSR relied on Chemical Abstracts Service (CAS) numbers that are unique numeric identifiers assigned to chemicals by the American Chemical Society.[9] Scientists consider

Table 1. Disclosed Use in Fracking of PFAS and Possible PFAS in New Mexico Oil and Gas Wells, 2013-2022

Chemical Name	Chemical Abstracts Service (CAS) Number	PFAS or PFAS Precursor?	Source of Determination as PFAS or PFAS Precursor
PTFE/Teflon	9002-84-0	PFAS	Identified as PFAS on EPA’s Master List of PFAS
Fluoroalkyl alcohol substituted polyethylene glycol	65545-80-4	PFAS	Identified as PFAS on EPA’s Master List of PFAS
Nonionic fluorosurfactant	Unknown (identity withheld as a trade secret)	Could be PFAS or PFAS precursor.	Some chemical experts identify nonionic fluorosurfactants as PFAS or PFAS precursors, others as likely to be PFAS or possibly PFAS.
Trade secret surfactants	Unknown (identity withheld as a trade secret)	Could include fluorosurfactants that are PFAS or PFAS precursors.	No determination possible where chemical identity is withheld.
Trade secret chemicals	Unknown (identity withheld as a trade secret)	Could include PFAS or PFAS precursors.	No determination possible where chemical identity is withheld.

This table shows the types of chemicals that are PFAS or could be PFAS that oil and gas companies injected for fracking into oil and gas wells in New Mexico between January 1, 2013 and September 29, 2022. PFAS precursors are chemicals that can break down into PFAS. Some scientists believe that if a chemical can break down into a PFAS, it could or should be considered a PFAS.[12]

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* Gas, the principal component of which is methane, is also known as “natural” gas, “fossil” gas and “fracked” gas.

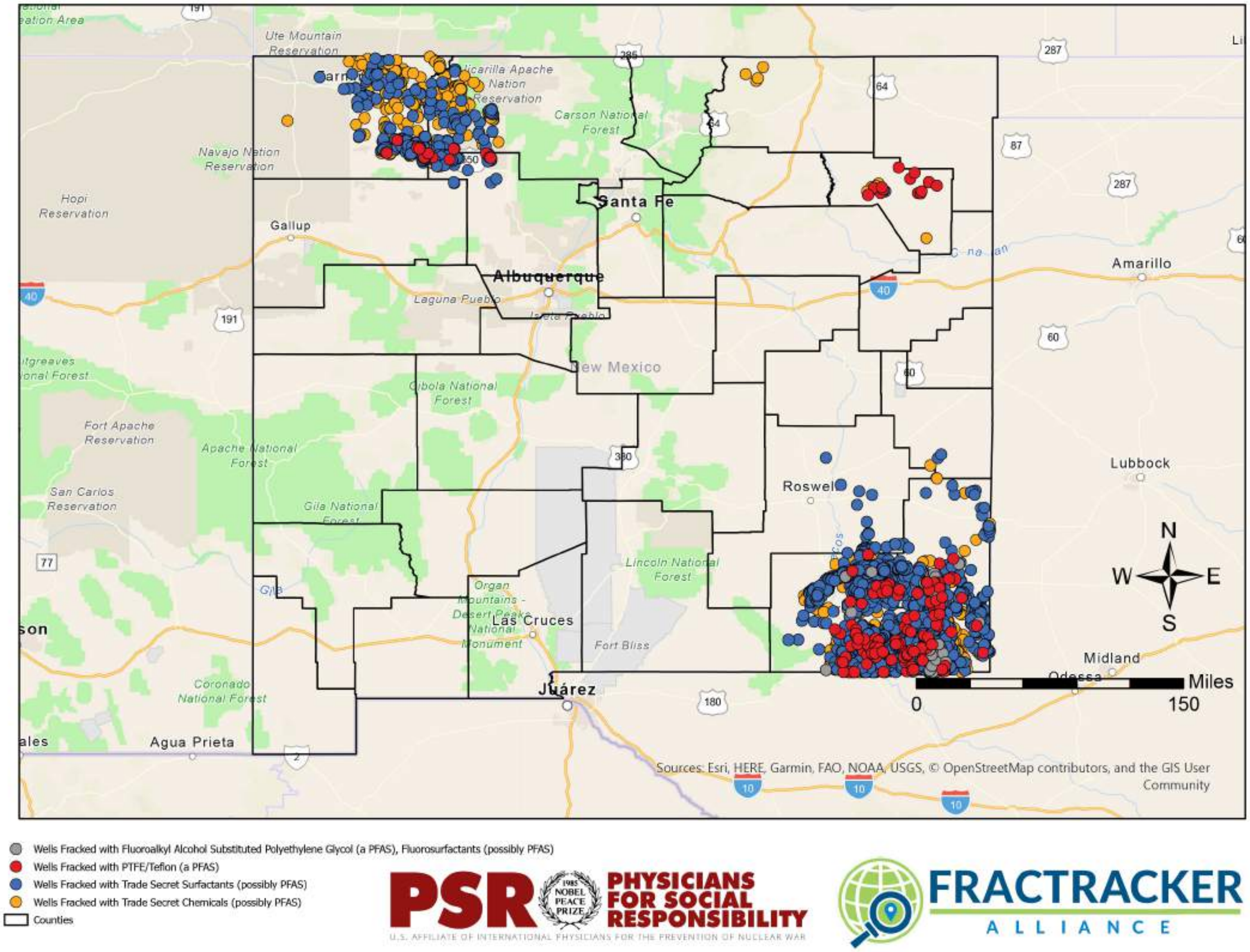
** In this report, the term “fracking” is used to discuss a particular stage in oil and/or gas production as distinct from other stages or methods of production such as drilling that precedes fracking. The terms “oil and gas production,” “oil and gas extraction,” and “oil and gas operations” cover the entire process of producing oil and/or gas.

CAS numbers the best way to identify chemicals because chemicals can have multiple names or trade names but only one CAS number.[10]

In addition, PSR found that oil and gas companies injected 24 wells in Eddy and Lea Counties with unspecified nonionic fluorosurfactants that could be PFAS or precursors (chemicals that could degrade into PFAS), according to three chemists and a board-certified toxicologist who reviewed the fluorosurfactants' names.[11]

The wells injected with PFAS or possible PFAS may significantly underrepresent the extent of PFAS use in the state's oil and gas wells, due to gaps in chemical disclosure

New Mexico Oil & Gas Wells Fracked with PFAS and Possible PFAS, Including Trade Secret Chemicals, 2013-2022



This map shows the location of oil and gas wells in New Mexico known to have been fracked between January 1, 2013 and September 29, 2022 using PTFE/Teflon (a known PFAS), fluoroalkyl alcohol substituted polyethylene glycol (a known PFAS), fluorosurfactants that may be PFAS or PFAS precursors, trade secret chemicals, and/or trade secret surfactants. An interactive version of the map is available at https://psr.org/new-mexico-pfas-map/. Users can zoom in to identify wells near them. For a more detailed explanation of data sources, see the Appendix.

rules, including those that allow oil and gas companies to conceal from the public as trade secrets the specific identities of chemicals they use in fracking. **PSR's review of fracking chemical disclosure in New Mexico found that oil and gas companies disclosed that they used fracking chemicals between 2013 and 2022 in 9,066 oil and gas wells. Of those wells, the companies injected more than 90 percent with at least one trade secret chemical and more than 40 percent with at least one trade secret surfactant. Some of these trade secret chemicals could be PFAS.**

The use of these chemicals is particularly alarming as New Mexico's oil production has increased seven-fold in roughly a decade, from about 65.5 million barrels in 2010 to more than 457 million barrels in 2021,[13] and gas production has roughly doubled from about a trillion cubic feet in 2013 to more than two trillion cubic feet in 2021.[14] While these increases, driven largely by production in the Permian Basin,[15] mean more revenue for the state,[16] they also mean more wells being drilled and fractured, more greenhouse gas emissions,[17] and more opportunities for drilling companies to use PFAS or other toxic chemicals.

b. Manmade and Dangerous: PFAS's History and Health Effects

PFAS are a class of thousands of synthetic chemicals manufactured to have properties that are valuable in multiple industrial contexts, including being slippery, oil- and water-repellant, and able to serve as dispersants or foaming agents.[18] PFAS have been called "perfluorinated chemicals" and "polyfluorinated compounds," or PFCs, though the term currently preferred by EPA is PFAS.[19]

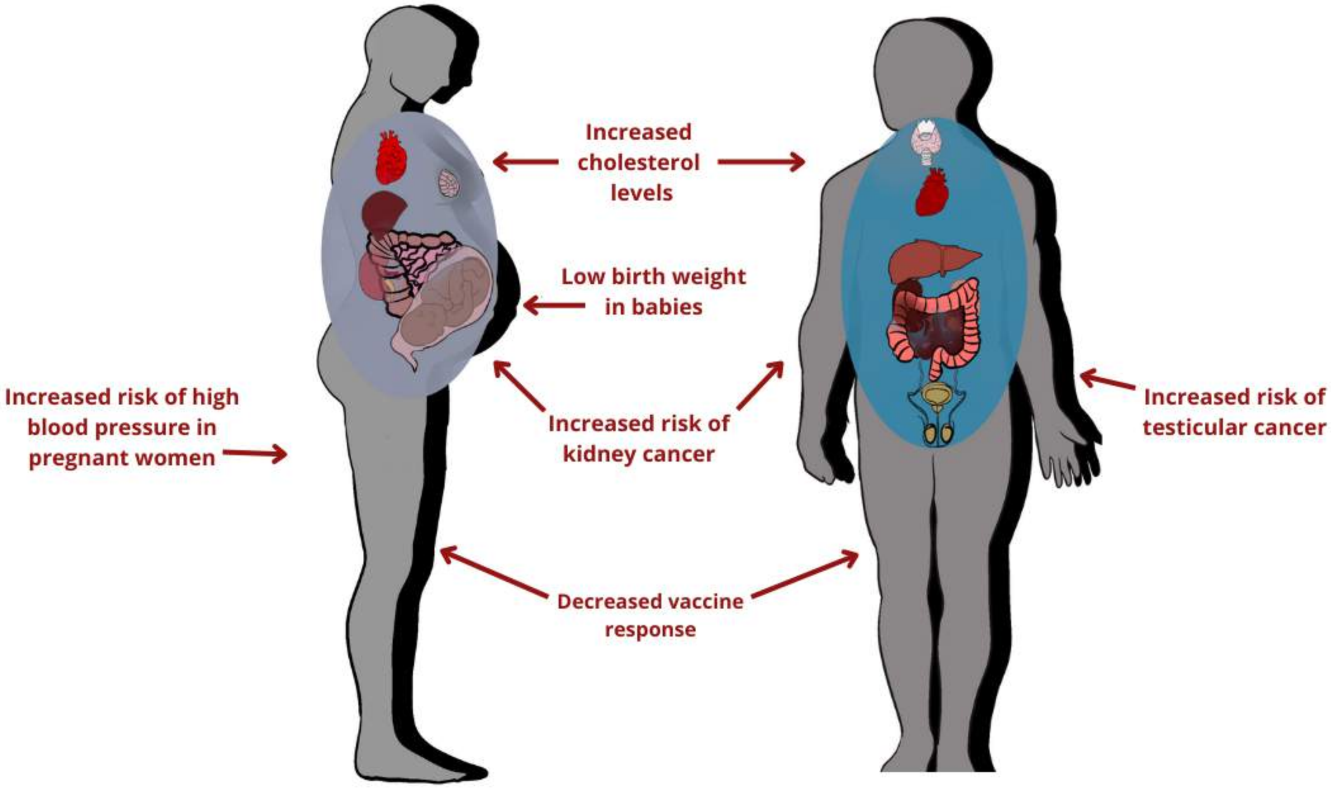
The first PFAS to be sold commercially was created by a chemist at Dupont and was patented as Teflon. Since 1949, it has been used in thousands of products, from nonstick cookware to waterproof clothing to plastics to dental floss.[20] Other PFAS chemicals, the most prominent of which are known as PFOA and PFOS, were used in food packaging, fire-fighting foam, and in 3M's widely used fabric protector, Scotchgard.[21] EPA reported in 2021 that about 650 types of PFAS remained in commerce.[22] Weak chemical disclosure laws make it difficult for the Agency to identify which PFAS chemicals are used, and where.

Between the 1960s and 1990s, researchers inside Dupont and 3M became aware that at least some of the PFAS they were manufacturing or using, particularly PFOA and PFOS, were associated with health problems including cancers and birth defects, had accumulated in people worldwide, and persisted in the environment.[23]

Many of these facts, kept internal by the companies, came to light after attorney Rob Bilott filed lawsuits in 1999 and 2001 accusing Dupont of causing pollution in and around Parkersburg, West Virginia with PFOA, a type of PFAS then used in making PTFE (Teflon).[24] In December 2011, as part of Dupont's settlement of the 2001 lawsuit, a team of epidemiologists completed a study of the blood of 70,000 West Virginians and found a probable link between PFOA and kidney cancer, testicular cancer, thyroid disease (over- or under-production of hormones by the thyroid gland), high cholesterol, pre-eclampsia (a potentially dangerous complication during pregnancy characterized by high blood pressure and signs of damage to other organ systems, most often the liver and kidneys), and ulcerative colitis (a disease causing inflammation and ulcers in the large intestine or colon).[25]

Current peer-reviewed scientific research on PFAS suggests that exposure to certain levels of some PFAS may lead to adverse health outcomes. Research findings differ, as different studies have examined different PFAS chemicals, different types or levels of exposure, or different exposed populations. However, some findings are more widely endorsed; for example, the U.S. Environmental Protection Agency (EPA)[26] and the Center for Disease Control and Prevention's Agency for Toxic Substances and Disease Registry (ATSDR)[27] agree that exposure to high levels of certain PFAS may lead to increased risk of high blood pressure in pregnant women; low birth weight in babies;

POTENTIAL HEALTH EFFECTS OF PFAS EXPOSURE



Exposure to PFAS chemicals can result in a variety of serious health effects including those indicated above.Source: U.S. Environmental Protection Agency, Agency for Toxic Substances and Disease Registry. Graphic by Astra Robles

increased risk of kidney or testicular cancer; decreased vaccine response, and increased cholesterol levels. Research is ongoing to determine the health effects of different levels of exposure to different PFAS, including the health effects of long-term, low-level PFAS exposure, especially in children. **See graphic above.**

PFAS are not only highly toxic; they also demonstrate extreme persistence in the environment. PFAS' nickname "forever chemicals" reflects their chemistry – created by chemical manufacturers – that features a bond between fluorine and carbon atoms that is among the strongest in chemistry and rarely if ever exists in nature. The result: chemicals that are extremely resistant to breaking down.[28] PFAS are also extremely mobile in water,[29] making them able to spread through the environment via groundwater or surface water. Another risk, discussed in Chapter 5, is that PFAS could compound the health effects from other dangerous chemicals associated with oil and gas production.

c. EPA Recognizes Risks of PFAS

EPA has been slow to regulate PFAS, but the agency has taken actions, particularly in recent years, that recognize

PFAS's extraordinary risks. In June 2022, reflecting growing public concern about PFAS, EPA significantly lowered its non-binding health advisory level for PFOA and PFOS in drinking water. Previously, EPA had set the combined health advisory level for these chemicals at 70 parts per trillion.[30] **"The new published peer-reviewed data and draft EPA analyses…" EPA wrote in June 2022, "indicate that the levels at which negative health outcomes could occur are much lower than previously understood."**[31] EPA set its new interim health advisory level for PFOA in drinking water to 0.004 parts per trillion and its interim health advisory level for PFOS to 0.02 parts per trillion.[32] EPA also set new final health advisory levels for two other PFAS known as GenX and PFBS at 10 parts per trillion and 2,000 parts per trillion, respectively.[33] EPA said that its interim health advisory levels were intended to provide guidance until enforceable drinking water regulations for PFAS take effect.[34]

EPA then in March 2023 released proposed standards for levels of six PFAS in drinking water. These included a level of four parts per trillion for both PFOA and PFOS. EPA explained in an email, sent in response to a question from PSR, the difference between the health advisory levels and the proposed drinking water standards:

> Health advisories reflect EPA's assessment of health risks of a contaminant based on the best available science and provide advice and information on actions that water systems may take to address contamination for these and other PFAS.[35]

Besides focusing on possible health effects, health advisories differ from rules in that they do not take into account whether a particular level of protection can be achieved or at what cost.[36] In this respect they resemble EPA's proposed Maximum Contaminant Level Goal, which for PFOA and PFOS is zero in drinking water.

EPA's interim health advisory levels mean that the toxicity of PFOA is almost beyond comprehension. According to EPA's advisory levels, one tablespoon of PFOA would be enough to contaminate 1.75 trillion gallons of water,[37] which is more than twice the total storage capacity of Elephant Butte Reservoir (720 billion gallons),[38] which forms New Mexico's largest lake on the Rio Grande River in the southwestern part of the state.[39] (Current levels in the lake are far below total storage capacity due to drought.[40]) EPA's new health advisory levels further show that PFOS is similarly extraordinarily toxic. In March 2023, EPA proposed drinking water regulations that would limit the amount of PFOA and PFOS in drinking water to four parts per trillion. The agency also proposed that drinking water providers limit the combined levels of four other types of PFAS: PFNA, PFHxS, PFBS, and/or GenX Chemicals. The agency said that it expects to finalize the rule by the end of 2023.[41]

Several experts told PSR that because of the extreme potency of certain types of PFAS and the fact that chemical makers have created thousands of these forever chemicals, they would recommend particular testing methods to detect PFAS in the environment. The scientists are Linda Birnbaum, Ph.D., D.A.B.T., A.T.S., a board-certified toxicologist and former director of the National Institute of Environmental Health Sciences;[42] Zacariah Hildenbrand Ph.D., research professor in Chemistry and Biochemistry at the University of Texas at El Paso;[43] Kevin Schug Ph.D., Shimadzu Distinguished Professor of Analytical Chemistry at the University of Texas at Arlington,[44] and Wilma Subra, holder of a master's degree in chemistry and recipient of a John D. and Catherine T. MacArthur Foundation "Genius" grant for her work helping to protect communities from toxic pollution.[45] All were in agreement in recommending the use of testing equipment that can detect PFAS in concentrations at least as low as single-digit parts per trillion. They further recommended testing for total organic fluorine in addition to testing for specific types of PFAS. Total organic fluorine is a marker that would indicate the presence of PFAS even if a specific PFAS were not tested for. Testing for specific PFAS only might fail to detect other forms of PFAS present in the sample.

d. PFAS Already Present in New Mexico's Environment

Evidence has mounted over the years of cases of PFAS pollution from a variety of sources, including in New Mexico.

In 2018, the U.S. Air Force reported that PFAS had been detected in groundwater below Cannon Air Force base in Clovis and beneath Holloman Air Force base outside Alamogordo.[46]

At Cannon Air Force base, the levels were as high as 26,200 parts per trillion in groundwater for combined PFOA and PFOS.[47] At Holloman Air Force base, the levels reached as high as 1,294,000 parts per trillion for combined PFOA and PFOS.[48] (As noted above, EPA's health advisory levels for PFOA and PFOS in drinking water are 0.004 parts per trillion and 0.02 parts per trillion, respectively.) In both cases, the pollution was linked to the use of firefighting foam that contained PFAS.[49] It is unclear whether both PFOA and PFOS were in the foam. The Interstate Technology Regulatory Council reports that firefighting foam used between the 1960s and 2002 can contain both types of PFAS as well as precursors that may degrade into PFOA.[50] The pollution near Cannon Air Force Base devastated a local dairy farm. Because of the pollution, farmer Art Schaap told the Albuquerque

Journal in 2022 that since he learned of the PFAS contamination in his water in 2018, he had been unable to sell his cow's milk or meat. He was forced to euthanize thousands of cows, and he and the state must determine how to safely dispose of the PFAS-tainted carcasses so that the persistent pollutants do not cause further contamination. "I've lost so much money, I don't know if I can restart," Schaap told the Journal.[51]

The state Environment Department began a water sampling effort in mid-2020 with support from the U.S. Geological Survey to determine levels of PFAS around the state, and the concentrations discovered showed some cause for concern.[52] The sampling, which ran from August 2020 to November 2021, focused on ground and surface water supplies in 19 New Mexico counties.[53] In a news release published in January 2021, the Department reported that "To date, the data from this effort does not indicate any imminent public health threats....None of the results received so far show levels of PFOS or PFOA at or above the [EPA's] Lifetime Health Advisory." However, that health advisory of 70 parts per trillion of combined PFOA and PFOS is now outdated. Under EPA's June 2022 interim health advisory levels for PFOA and PFOS, multiple samples of water in New Mexico's sampling for PFAS have levels that are now judged unsafe. They range from 145 times to 9,000 times EPA's interim health advisory levels for PFOA and PFOS, including:

- 2.9 parts per trillion of PFOS in the Melrose water system in Curry County (145 times EPA's interim health advisory level);

- 8 parts per trillion of PFOA in the Alamogordo Domestic Water System/Golf Course Well in Otero County (2,000 times EPA's interim health advisory level), and

- 36 parts per trillion of PFOA in spring 10 of the Cloudcroft Water System in Otero County (9,000 times EPA's interim health advisory level).

"If, during the study, levels of PFOS and PFOA are detected in drinking water resources above the Lifetime Health Advisory," the department wrote, "NMED will work with public water systems to identify the best mitigation options, if requested." It is unclear if the department will take the same steps if the levels detected are above EPA's much more protective interim health advisory levels. The department has said on a separate website that "PFAS contamination in New Mexico is one of the New Mexico Environment Department's top priorities, as is the protection of human health and the environment."[54] The Department added that in the absence of federal drinking water standards for PFAS, it was considering developing standards of its own.[55]

e. Oil and Gas Operations Provide Many Potential Routes of Exposure to PFAS

Oil and gas operations in New Mexico deserve scrutiny as a possible additional source of PFAS contamination due to the now-documented use of PFAS in the state's oil and gas wells and the potential that people could be exposed to PFAS via multiple pathways.

An oil and/or gas site in Aztec, New Mexico, Sept. 2008. Photo credit: Jane Pargiter, EcoFlight.

EPA in its 2016 national report on fracking and drinking water found that fracking-related pollution could follow a number of pathways that could impact surface- and groundwater. The agency cited the following possible pathways to exposure:

- spills of fracking fluid that seep into groundwater;
- injection of fracking fluid into wells with cracks in the casing or cement, allowing the fluid to migrate into aquifers;
- injection of fracking fluids directly into groundwater;
- underground migration of fracking fluids through fracking-related or natural fractures;
- intersection of fracking fluid with nearby oil and gas wells,
- spills of wastewater after the fracking process is completed, and
- inadequate treatment and discharge of fracking wastewater to surface water supplies.[56]

PFAS used in oil and gas extraction could pollute water through any of these pathways, plus other routes discussed in more detail in Chapter 5 including through airborne releases and disposal of oil and gas wastewater in underground injection wells, a pathway that EPA did not examine in its 2016 report.[57]

PFAS contamination could further reduce available water supplies. EPA reported in 2013 that "about 87 percent of New Mexico's public water supply comes from groundwater. No other southwestern state gets such a large percentage of its domestic water from groundwater sources."[58] A representative of the federal Bureau of Reclamation told the Carlsbad Current-Argus in 2021 that in the Pecos River Basin in southeastern New Mexico, 80 percent of water was consumed by agricultural interests for irrigation, and 64 percent of that water came from groundwater. Much of the Pecos Basin overlaps with the heavily drilled Permian Basin.[59] The EPA stated in 2015 that "because groundwater usually moves slowly, contaminants generally undergo less dilution than when in surface water."[60] The agency added that

> [b]ecause ground water generally moves slowly, contamination often remains undetected for long periods of time. This makes cleanup of a contaminated water supply difficult, if not impossible. If a cleanup is undertaken, it can cost thousands to millions of dollars.[61]

Furthermore, water supplies are expected to shrink in future years as the climate heats up, making clean water supplies even more important. The Bureau of Reclamation forecast that in coming years, farmers in the basin will encounter higher temperatures and scarcer water.[62] PFAS contaminate could further reduce available water supplies.

f. PFAS: Among Many Dangerous Chemicals Used in Fracking

When used in oil and gas operations, PFAS may add to the cumulative human exposure to a host of toxic substances. In the fracturing stage of oil and gas production, chemicals serve a variety of purposes including killing bacteria inside the wellbore, reducing friction during high-pressure fracking, and thickening the fluid so that the sand, suspended in the gelled fluid, can travel farther into underground formations.[63] In its 2016 study of fracking and drinking water, the EPA identified 1,606 chemicals used in fracking fluid and/or found in fracking wastewater. While the agency found high-quality information on health effects for only about 10 percent (173) of these chemicals, that information was troubling. EPA found that health effects associated with chronic oral exposure to these chemicals include carcinogenicity, neurotoxicity, immune system effects, changes in body weight, changes in blood chemistry, liver and kidney toxicity, and reproductive and developmental toxicity.[64]

Chemicals used in the drilling stage that precedes actual fracturing can also pose health risks, including developmental toxicity and the formation of tumors, according to EPA regulators.[65] A disclosure form filed with the state of Ohio, one of only two states to require public disclosure of drilling chemicals (Colorado is the other),[66] shows that Statoil, Norway's state oil company (since renamed Equinor), has used the neurotoxic chemical xylene in drilling.[67] In short, when chemicals used in drilling, fracking or other stages and methods of oil and gas operations come into contact with people or the environment, they can produce serious negative health effects.[68]

CH. 2 PFAS USED IN NEW MEXICO’S OIL AND GAS WELLS: A DEEPER LOOK

a. PTFE (Teflon), a PFAS Fluoropolymer

One of the types of PFAS used for fracking in New Mexico’s oil and gas wells between 2013 and 2022 was PTFE, commonly known as Teflon.

PTFE is a fluoropolymer, a type of plastic.[69] Scientists’[70] and environmentalists'[71] major concerns about PTFE and other fluoropolymers are related less to these substances themselves, but rather to the associated impacts of their production, use, and disposal. The production of PTFE and other fluoropolymers relies on the use of other, highly toxic PFAS that are used as production aids. As noted in a peer-reviewed study published in 2020, these other PFAS have included fluorosurfactants such as PFOA, whose risks are discussed in the previous chapter, and GenX, which is similarly harmful and has replaced PFOA in fluoropolymer production.[72] PTFE and other fluoropolymers may contain these more toxic PFAS fragments, and those fragments may leach out of the PTFE during use.[73] The authors of the 2020 paper noted that

> The levels of leachables…in individual fluoropolymer substances and products depend on the production process and subsequent treatment processes; a comprehensive global overview is currently lacking.[74]

In addition, PTFE may generate other PFAS if the PTFE breaks down under heat.[75]

The 2020 paper authors noted that the persistence in the environment of PTFE and other fluoropolymers could pose problems during disposal, observing that “Landfilling of fluoropolymers leads to contamination of leachates with PFAS and can contribute to release of plastics and microplastics.[76] One of the authors added in an email to PSR that if PTFE were used in oil and gas wells that have especially high temperatures, defined in publications by oilfield services company, Schlumberger, as 300º-350º F or higher for so-called “high-pressure, high-temperature wells,”[77] the PTFE could undergo a process called “thermolysis” and generate toxic PFAS called perfluoroalkyl carboxylic acids (PFCAs). As a result, he wrote, “there could be some additional problems that need some investigation.”[78] A representative from New Mexico’s Oil Conservation Division said that wells with the characteristics described by Schlumberger “would be atypical for any oil or gas producing wells in New Mexico.” He added that the Oil Conservation Division does not track pressures or temperatures inside oil and gas wells, though operators sometimes report downhole pressures during initial production testing or “may report the temperatures in the well logs.”[79] These data are publicly accessible online.[80]

In 2021, a coalition of national environmental organizations including the Center for Environmental Health, Clean Water Action, Ecology Center, Environmental Working Group, Natural Resources Defense Council, Safer States, and the Sierra Club voiced several environmental and health concerns regarding the risks of fluoropolymers such as

Table 2. Disclosed Use in Fracking of PTFE in New Mexico Oil and Gas Wells, 2013-2022

County Name	Number of wells injected with PTFE	Mass of PTFE (lbs.)[84]
Eddy	113	2028
Harding	14	2
Lea	74	557
Rio Arriba	2	2
San Juan	18)	10
Sandoval	6	6
Total	**227**	**2605**

This table shows by county the number of New Mexico wells in which oil and gas companies injected PTFE for fracking between 2013 and 2022. For a more detailed explanation of data sources, see the Appendix.

PTFE, based on their review of multiple scientific articles. The groups also noted that fluoropolymers are manufactured with chemicals that have an outsized negative effect on climate change.[81]

Public records make it difficult to know for what purpose PTFE was used. In most cases, either no purpose or various purposes were listed for chemical products, but the individual chemical components of these products were listed in a separate portion of the disclosure form, making it impossible to know which components are part of which product.[82] However, PTFE, which is marketed as Teflon, is known for its slipperiness, suggesting it might have been used as a friction reducer, a common purpose for fracking chemicals.[83]

Oil and gas companies that have disclosed using PTFE for fracking in New Mexico (Table 3) include ExxonMobil Corp., the nation's largest publicly traded oil and gas company;[85] and Devon Energy Corp.[86] and Occidental Petroleum Corp.,[87] both major producers in the Permian Basin.

Disclosure gaps in New Mexico law, discussed below, may prevent scientists and the public from knowing the extent of the use of PTFE and other PFAS in the state's oil and gas operations.

b. Fluoroalkyl Alcohol Substituted Polyethylene Glycol

The other type of PFAS disclosed as being used for fracking in New Mexico's oil and gas wells between 2013 and 2022 was fluoroalkyl alcohol substituted polyethylene glycol. EOG Resources, a major oil producer in the Permian and San Juan Basins,[88] injected 34 wells, all in Lea County, with a total of 6,400 pounds of this chemical. Fluoroalkyl alcohol

Table 3. Oil and Gas Companies that Fracked Wells in New Mexico Using PTFE, 2013-2022

Well Operator	Number of wells injected with PTFE	Total mass of PTFE (lbs.)
Devon Energy Production Company L. P.	60	456
Occidental Oil and Gas	45	354
Matador Production Company	23	204
Yates Petroleum Co.	22	No data available
Cimarex Energy Co.	13	134
Encana Oil & Gas Inc.	12	20
Whiting Petroleum	10	1
WPX Energy	9	No data available
XTO Energy/ExxonMobil	7	1286
BreitBurn Operating LP	6	2
ConocoPhillips Company/Burlington Resources	4	30
Energen Resources Corp.	3	No data available
COG Operating LLC	2	10
Dugan Production Corp.	2	No data available
Kaiser-Francis Oil Company	2	No data available
BOPCO, L.P.	1	No data available
DGP Energy	1	14
Mewbourne Oil Co.	1	No data available
Murchison Oil and Gas Co.	1	7
Oxy USA Inc.	1	No data available
Tap Rock Resources	1	90
V-F Petroleum Inc.	1	No data available

This table shows the oil and gas companies that fracked oil and gas wells in New Mexico with PTFE between 2013 and 2022. For a more detailed explanation of data sources, see the Appendix.

substituted polyethylene glycol is listed on EPA's Master List of PFAS Substances under a different name.[89] PSR was able to identify it there using its CAS number of 65545-80-4, which appears in the FracFocus records.[90] Its purpose as declared in FracFocus is "oil field surfactant," suggesting that it could be a fluorosurfactant,[91] a type of chemical discussed in more detail in Chapter 3.

Limited toxicological data is available about chemical 65545-80-4, but according to data on the website of the National Library of Medicine's ChemIDplus, at high doses, the chemical is associated with convulsions or effects on the threshold for seizures; dyspnea, or shortness of breath; and muscle weakness.[92] A safety data sheet for the chemical published by its manufacturer says little about human health effects. "To the best of our knowledge," the safety data sheet says, referencing the substance using a trade name Zonyl® FSO-100, "the chemical, physical, and toxicological properties have not been thoroughly investigated."

Regarding impacts to the environment, the safety data sheet says, "Toxic to aquatic life with long lasting effects… Avoid release to the environment…Collect spillage…Dispose of contents/ container to an approved waste disposal plant."[93] A message on the website of ChemPoint, a chemical distributor, suggests that this chemical was phased out due to concerns that it could break down into PFOA or PFOS. A message apparently from Chemours, a company spun off from Dupont, says

> Zonyl® fluorosurfactant and repellent grades were discontinued between 2009 and 2014. Capstone® fluorosurfactants [a new type of fluorosurfactant] and repellents were introduced as sustainable replacements that meet the goals of the U.S. EPA 2010/15 PFOA Stewardship Program. They are based on short-chain molecules that cannot break down to PFOA or PFOS in the environment."[94]

However, as is discussed below, scientists have raised concerns about the health and environmental effects of these replacement chemicals.

Table 4. Disclosed Use of Fluoroalkyl Alcohol Substituted Polyethylene Glycol in New Mexico Oil and Gas Wells, 2013-2022

Well Operator	**Number of wells injected with fluoroalkyl alcohol substituted polyethylene glycol – all in Lea County**	**Total weight of fluoroalkyl alcohol substituted polyethylene glycol (lbs.)**
EOG Resources, Inc.	34	6,400

This table shows that EOG Resources, Inc., fracked oil and gas wells in New Mexico with fluoroalkyl alcohol substituted polyethylene glycol between 2013 and 2022. For a more detailed explanation of data sources, see the Appendix.

CH. 3

NEW MEXICO'S CHEMICAL DISCLOSURE LAWS SHIELD CHEMICAL IDENTITIES

a. New Mexico's "Trade Secret" Law Shields Potentially Dangerous Substances, Including PFAS

The danger of exposure to unknown chemicals – PFAS and others – from oil and gas operations persists in New Mexico, despite state rules that generally require public disclosure of fracking and drilling chemicals.[95] On the face of it, these disclosure requirements seem effective. However, an important exception allows companies to avoid full and meaningful disclosure: The law allows chemical manufacturers, well operators and other companies in the chemical supply chain to withhold exact fracking and drilling fluid ingredient information if they deem it a trade secret.** [96] In some cases in New Mexico fracking chemical disclosure records, oil and gas operators disclose generic names of chemicals while withholding as trade secrets their specific identities. These generic identifiers include "nonionic fluorosurfactant,"[97] a chemical identified as PFAS or possible PFAS by several scientists as discussed below, and "proprietary Acid Inhibitor/Surfactant."[98] Regrettably, the use of such vague descriptors can hide from public view the true identities of dangerous chemicals, including PFAS. The use of trade secrets to conceal chemicals' specific identities effectively undermines the public health benefits of disclosure by preventing health professionals, first responders, state regulators and the public from knowing where PFAS – or other toxic chemicals – have been used in oil and gas wells.

In addition to allowing trade secret exemptions, New Mexico does not require public disclosure of chemicals used in drilling, enhanced oil recovery, or in other extraction techniques that are distinct from fracking per se. Chemicals used during the first stage of the drilling process would be highly likely to leach into groundwater since during this stage, according to EPA, drilling passes directly through groundwater zones[99] before any casing or cement is placed in the well to seal it off. The resulting potential for groundwater contamination makes public disclosure of chemicals used in drilling especially important, as these regulatory gaps increase the potential that New Mexicans could unknowingly be exposed to PFAS and other chemicals used during oil and gas extraction.[100]

In at least some cases, the New Mexico Oil Conservation Division has prohibited oil and gas companies from using "oil base muds" for drilling "until fresh water zones are cased and cemented providing isolation from the oil or diesel. This includes synthetic oils."[101] Such "muds," according to oilfield services company Schlumberger, are "generally synonymous with drilling fluid."[102] According to the Oklahoma State University Extension Service, oil-based muds can include diesel fuel and the highly dangerous chemicals benzene, toluene, ethylbenzene, and xylene.[103]

It is unclear whether New Mexico's prohibition would prohibit the use of PFAS during drilling that passes through fresh water zones.

b. Extensive Use of "Trade Secret" Claims Veils Actual Use

PSR found extensive application of the trade secret provisions under New Mexico's fracking chemical disclosure rules – so extensive that it could serve to mask widespread use of PFAS in the state's oil and gas wells. Our data analysis revealed that, between 2013 and 2022, New Mexico's well operators claimed at least one fracking chemical as a trade secret in 8,293 oil and gas wells located across 11 counties. The trade secret chemicals used in New Mexico over this roughly 10-year period totaled 243 million pounds (see Table 5).[104] If even a small fraction of this weight were PFAS, that fraction could pose significant risks to health and the environment. In an effort to identify PFAS among these trade secret chemicals, PSR examined whether any were listed as a surfactant or a fluorosurfactant. According to EPA, surfactants are commonly used in fracking[105] and lower the surface tension of a liquid, the interaction at the surface between two liquids (called interfacial tension), or

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** Trade secret information is also called "proprietary" or "confidential business information" (CBI).

the interaction between a liquid and a solid.[106] Compared to other surfactants, fluorosurfactants are said to be "superior in their aqueous surface tension reduction at very low concentrations and are useful as wetting and leveling agents, emulsifiers, foaming agents, or dispersants."[107] At least some fluorosurfactants are PFAS, including the dangerous chemicals PFOA and PFOS[108] and 8:2 fluorotelomer alcohol,[109] a nonionic fluorosurfactant[110] that can break down into PFOA.[111] Two scientists told PSR that all or most fluorosurfactants could be classified as a PFAS[112] while two other scientists were uncertain.[113]

Like the broader class of surfactants, fluorosurfactants are also used in fracking, and perhaps other stages and methods of oil and gas extraction, according to scientific and industry sources. In 2020, several scientists published an article in *Environmental Science: Processes and Impacts* showing that since 1956, PFAS including fluorosurfactants had been used or proposed to be used globally in oil and gas extraction techniques including chemical-driven gas production, chemical flooding, fracking, and the drilling that precedes fracking and other oil and gas production techniques.[114] In 2008, two authors, one of whom was identified as an employee at DuPont, wrote in the peer-reviewed *Open Petroleum Engineering Journal* that the use of fluorosurfactants was relatively common in the oil and gas industry and that their use was about to surge. They referred to fluorosurfactants as an "emerging technology" and stated,

> While fluorosurfactants have been used in gas and oil exploration for four decades, the increased demand for petroleum and the greater understanding of the benefits of fluorosurfactants have led to growing acceptance for fluorosurfactants throughout the petroleum industry.[115]

The authors did not explicitly say that fluorosurfactants used in oil and gas operations were PFAS but they described the fluorosurfactants in ways that are commonly used to describe PFAS. They wrote that "The use of fluorosurfactants is a recent but growing trend due to (i) the exceptional hydrophobic [water-repellent] and oleophobic [oil-repellent] nature of the perfluoroalkyl and perfluoroalkyl ether groups...The bond strength of the carbon-fluorine bond in perfluoroalkyl and perfluoroalkyl ether groups has been demonstrated as the key to remarkable overall stability for fluorochemicals and fluoropolymers."[116] This evidence suggests that any time an unidentified surfactant or fluorosurfactant is used in oil and gas production, there is a potential that it is a PFAS.

We found thousands of cases of oil and gas companies using at least one trade secret chemical that they described as a surfactant. These occurred in 3,680 wells, spread across 10 counties (see Table 5).[117] Operators' names for these chemicals were vague, including "surfactant" and "surfactant blend." These trade secret surfactants totaled 19.3 million pounds. (See examples from individual wells in Table 8 below.) While we cannot know what these trade secret chemicals are, should even a small percentage of them be fluorosurfactants that are PFAS, they could pose significant threats to human health and the environment.

In 24 wells (16 in Eddy County and 8 in Lea County), oil and gas companies disclosed the use of trade secret chemicals listed with the nonspecific name "nonionic fluorosurfactant" that are apparently fluorosurfactants and may be PFAS. The weight of these chemicals totaled 970 pounds.[118] Even if some of that volume were PFAS, it could pose significant health and environmental risks, depending on the chemicals' toxicity. According to two Texas university-based chemists, Hildenbrand and Schug, both of whom are authors of multiple peer-reviewed articles about chemicals related to oil and gas production,[119] nonionic fluorosurfactants are PFAS or could degrade into PFAS. In addition, Subra, the chemist and MacArthur Foundation "Genius" grant winner, identified the chemicals as potential PFAS.[120] Still another expert, toxicologist Birnbaum, informed PSR that the chemicals are likely to be PFAS.[121] Birnbaum added that PFAS, perhaps including the nonionic fluorosurfactants used in New Mexico's oil and gas wells, could degrade into one or more smaller PFAS[122] (Hildenbrand agreed). Birnbaum,[123] Hildenbrand,[124] Subra,[125] and Schug[126] generally agree that if a chemical can break down into a PFAS, it could or should be considered a PFAS.

PSR has had to rely on scientists to identify these chemicals as PFAS, potential PFAS, or PFAS precursors because the oil and gas companies that made the public disclosures to FracFocus withheld as trade secrets the chemicals' CAS numbers, data that would have enabled a precise identification of the chemicals. The identification in the FracFocus records included only the generic name "nonionic fluorosurfactant" and the trade name "S-222" for the product containing the nonionic fluorosurfactants,[127] information insufficient to identify the chemicals with specificity. The fact that only one trade name was listed each time the chemical was reported suggests that the fluorosurfactant might be the same chemical in each use, but it is impossible to know without a CAS number. The sole purpose for which these chemicals were listed: "Surfactants."[128] The locations of the wells where nonionic fluorosurfactants were used are displayed in the map on page two.

Table 5. Disclosed Use of Trade Secret Chemicals in New Mexico Oil and Gas Wells, 2013-2022

County Name	No. of wells injected with at least one trade secret chemical	Mass of all trade secret chemicals (lbs.)	No. of wells injected with trade secret surfactants	Mass of trade secret surfactants (lbs.)	No. of wells injected with nonionic fluoro-surfactants	Mass of nonionic fluoro-surfactants (lbs.)
Chaves	62	2,590,000	41	174,000	0	0
Colfax	4	615	0	0	0	0
De Baca	1	1,490	1	273	0	0
Eddy	3,787	110,000,000	1,895	9,120,000	8	106
Harding	15	2,820	3	33	0	0
Lea	3,606	120,000,000	1,435	8,270,000	16	860
McKinley	2	397	2	11	0	0
Rio Arriba	271	1,980,000	68	138,000	0	0
Roosevelt	5	15,000	2	12,300	0	0
San Juan	415	5,200,000	179	1,140,000	0	0
Sandoval	125	2,590,000	55	415,000	0	0
Total	**8,293**	**243,000,000**	**3,681**	**19,300,000**	**24**	**966**

This table shows by county the number of New Mexico wells in which oil and gas companies injected at least one trade secret fracking chemical, at least one trade secret surfactant, and/or at least one unspecified nonionic fluorosurfactant. It also shows the total combined weight of these chemicals by county and statewide. The total weight figures reflect the sum of all records for which we have enough information to calculate a chemical's weight. However, the total weight figures represent an undercount because many fracking chemical disclosures lack sufficient data to perform this calculation. The wells injected with trade secret surfactants are a subset of the wells injected with trade secret chemicals. The wells injected with unspecified nonionic fluorosurfactants are a subset of the wells injected with trade secret chemicals and trade secret surfactants. For a more detailed explanation of data sources, see the Appendix.

Data show that multiple oil and gas companies have injected oil and gas wells in New Mexico with trade secret chemicals that could be or could break down into PFAS. The excerpted table below shows the 15 companies that fracked the most wells in New Mexico between 2013 and 2022 with at least one trade secret chemical.

Table 6. Excerpt (full table in Appendix). Oil and Gas Companies that Fracked the Most Wells in New Mexico Using Trade Secret Chemicals and Trade Secret Surfactants, 2013-2022

Operator	Number of wells injected with trade secret chemicals	Number of wells injected with trade secret surfactants
EOG Resources, Inc.	1177	214
COG Operating LLC	844	438
Devon Energy Production Company L. P.	586	358
Mewbourne Oil Company	575	116
Occidental Oil and Gas	498	141
XTO Energy/ExxonMobil	442	203
Apache Corporation	439	386
Cimarex Energy Co.	336	186
Matador Production Company	288	63
Chevron USA Inc.	264	189
Hilcorp Energy Company	203	0
ConocoPhillips Company/Burlington Resources	161	112
WPX Energy	148	21
Kaiser-Francis Oil Company	131	67
Lime Rock Resources Ii-A, L.P.	129	91

This excerpted table shows the oil and gas companies that fracked the greatest number of oil and gas wells in New Mexico with trade secret chemicals and trade secret surfactants between January 1, 2013 and September 29, 2022. The full table showing all of the companies that fracked at least one well with trade secret chemicals and trade secret surfactants between January 1, 2013, and September 29, 2022, is located in the appendix. The wells injected with trade secret surfactants are a subset of the wells injected with trade secret chemicals. For a more detailed explanation of data sources, see the Appendix.

Erratum: The heading for the middle column in Table 6 on page 15 was corrected to show that the numbers in that column reflect the number of wells injected with trade secret chemicals, 2013-2022.

Table 7. Oil and Gas Companies that Fracked Wells in New Mexico Using Nonionic Fluorosurfactants, 2013-2022

Well Operator	Number of wells injected with nonionic fluorosurfactants	Total weight of fluorosurfactants (lbs.)
Chevron USA Inc.	11	46
Apache Corporation	5	90
XTO Energy/ExxonMobil	4	814
COG Operating LLC	2	16
Nadel and Gussman Permian, LLC	1	<1
Seely Oil Co.	1	ND

This table shows the oil and gas companies that fracked oil and gas wells in New Mexico with unspecified nonionic fluorosurfactants between January 1, 2013 and September 29, 2022. The wells injected with the unspecified nonionic fluorosurfactants are a subset of the wells injected with trade secret chemicals and the wells injected with trade secret surfactants. For a more detailed explanation of data sources, see the Appendix.

ND=No Data Available

c. Examples of Individual Wells Injected with PFAS, Trade Secret Chemicals

Industry-disclosed data have allowed PSR to identify multiple types of fracking chemicals, including trade secret substances, that are injected into individual wells, as well as the quantities used. In some cases, oil and gas companies injected hundreds or even thousands of pounds of PFAS or trade secret chemicals into oil and gas wells for fracking. If the toxicities of some of these chemicals were similar to those of PFOA or PFOS, these quantities would be enough to contaminate vast amounts of water. Table 8 provides examples of the chemicals reported in several New Mexico wells.

Table 8. Examples of Chemical Reporting on Individual Oil and Gas Wells in New Mexico

Well Operator	Well Number	County	Year Fracking Completed	Chemical as Identified	CAS Number	Trade Name	Mass (lbs.)
EOG Resources, Inc.	3002542386	Lea	2015	fluoroalkyl alcohol substituted polyethylene glycol	65545-80-4	Plexflow RTS	120
XTO Energy/ ExxonMobil	3002542709	Lea	2015	nonionic fluorosurfactant	trade secret	S-222	226
XTO Energy/ ExxonMobil	3001542928	Eddy	2018	PTFE	9002-84-0	not reported	394
DJR Operating, LLC	3004321335	Sandoval	2020	surfactant 1	trade secret	FN2-02	29,400
Apache Corporation	3001545800	Eddy	2021	Surfactant Blend	trade secret	FRAQ SLIQ PFR-5560	4,559

This table shows illustrative samples of specific oil and/or gas wells injected with the types of fracking chemicals referenced in the larger tables above, including the identified PFAS fluoroalkyl alcohol substituted polyethylene glycol, fluorosurfactants, the identified PFAS PTFE, and trade secret surfactants such as “surfactant 1.” The examples cover a range of years and represent wells fracked in several New Mexico counties. For a detailed explanation of data sources, see the Appendix

CH. 4

EXPOSURE PATHWAYS TO PFAS ASSOCIATED WITH OIL AND GAS OPERATIONS IN NEW MEXICO

a. Evidence of Oil and Gas Drilling-Related Spills

The potential in New Mexico for water contamination from PFAS or other chemicals used in oil and gas operations is not just hypothetical. In 2017, the news outlet EnergyWire reported on spills at oil and gas sites in New Mexico and other states that had occurred over a five-year period. EnergyWire found 847 reported spills in New Mexico in 2012, 777 in 2013, 1,303 in 2014, 1,471 in 2015, and 1,311 in 2016.[129] According to the Center for Western Priorities, oil and gas companies operating in New Mexico reported 1,368 liquid spills in the state in 2021. The total volume spilled in 2021 was more than 4.7 million gallons, of which more than four million gallons was "produced water."[130] The remaining roughly 660,000 gallons was oil. New Mexico considers produced water to be a mixture that flows out of oil and gas wells, made up of the naturally occurring water from underground and "flowback" or wastewater from drilling and/or fracturing injected into the well that returns to the surface.[131] As such, produced water in New Mexico could contain PFAS or other man-made chemicals added to drilling and/or fracking fluid as well as naturally occurring contaminants found in the formation water such as radioactive substances.[132]

The EPA has indicated that oil can also contain residues of chemicals used in oil wells.[133] Therefore, it is possible that spills of produced water or oil could contain PFAS, even small amounts of which could cause significant and dangerous contamination. A review of New Mexico Oil Conservation Division records by the Center for Biological Diversity and WildEarth Guardians found that the number of spills reported in 2022 increased to more than 1,450.[134]

In 2019, a well operated by Enduring Resources, located in the exterior boundaries of the Counselors Chapter of the Navajo Nation Government,[135] spilled almost 60,000 gallons of oil and oil and gas wastewater.[136] A report prepared by a consultant for Enduring Resources found that the spill entered two tributaries of Escavada Wash and that groundwater in the area is less than 50 feet below the ground surface.[137] The New Mexico Bureau of Geology & Mineral Resources suggests that a wash is a wide, shallow streambed that is dry most of the time and that washes are similar to arroyos.[138] A state report found that the spill impacted groundwater or surface water – the report did not specify which type.[139] A 2018 report from the New Mexico Bureau of Geology and Mineral Resources suggests that the spill would have been likely to contaminate groundwater in part because "it is considered that a depth-to-water of less than 50 ft has high susceptibility" to contamination from oil and gas-related spills and because "[a]rroyo and valley bottoms are uniformly considered to be high susceptibility" for groundwater contamination following such spills.[140]

One particularly high-profile spill occurred in January 2020 when a pipeline carrying produced water burst at night, awakening Penny Aucoin and her husband Carl George and showering their home in Otis, New Mexico with wastewater for an hour. Aucoin told the NM Political Report that she was forced to euthanize 18 chickens and a dog and give up her remaining goat. She added that a county official informed her that she could not eat her chicken eggs or the chickens' meat and that she probably should avoid eating anything grown on her property. She and her husband reached a settlement[141] with the company that owned the pipeline, WPX Energy,[142] but Aucoin said that she remained concerned. She said during a news conference in January 2021,

> The dispute has been resolved amicably, but what scares me now is that people are blissfully unaware of the dangers that come with fracking, including the enormous amount of flow back waste [produced water] produced during the fracking process.

Aucoin said that she would be moving out of the area.[143]

b. Disposal of Wastewater Raises Pollution Concerns

The risk that PFAS and other chemicals could pollute the environment through the disposal of produced water is especially high because of the huge volumes involved. State data show that in 2022, the volume of produced water from New Mexico's oil and gas wells was almost 85 billion gallons, up from 67 billion gallons in 2021, and 57 billion gallons in 2020.[144]

According to a presentation by the state Environment Department, as of 2019, the most common method of produced water disposal was underground injection into wells that carry the wastewater into "deep, isolated geologic formations."[145] About 10 percent of the produced water was reused in oil and gas fields, where wastewater from oil and gas wells can be injected into oil wells to facilitate oil production in a process known as enhanced oil recovery or EOR. The surge of drilling in the Permian Basin has increased the generation of produced water and the need for more underground injection wells. Earthworks reported that

> [a]s of December, 2019, New Mexico had 983 active Class II disposal wells and 3,249 Class II EOR wells, for a total of 4,232. With the rapid expansion of Permian Basin development, the number of injection well permit applications has dramatically risen over time, with 538 new applications in 2019.[146]

If even a small percentage of the staggering amount of wastewater injected underground were tainted with PFAS, it could create significant pollution should it enter groundwater or surface water.

That fear is not unfounded; researchers have known for decades that produced water from injection wells can contaminate groundwater. In some cases, the produced water has migrated upward from deep underground, moving through nearby oil and gas wells, many of which have ceased operating but have not been properly sealed off from the surrounding underground rock formations.[147] This migrating wastewater can break out of abandoned wells and contaminate groundwater near the earth's surface.[148] In 1985, the Texas Department of Agriculture reported that it had a name for this phenomenon: "saltwater breakout," a reference to the high salt content of produced water.[149] The department quoted the Congressional Office of Technology Assessment regarding the "insidious" problem of underground injection of oil and gas wastewater. The Congressional office noted that such wastewater is typically injected in exactly the places where prior drilling has created opportunities for the wastewater to migrate into groundwater.[150] The department further reported that produced water could contaminate groundwater through leaks in an injection well's steel or cement casing, designed to seal the well off from groundwater supplies.[151] The consequences of such events are particularly acute in New Mexico with its heavy reliance on groundwater.

In 1989, Congress' investigative arm, the General Accounting Office (now the Government Accountability Office) found multiple cases of water contamination linked to underground injection wells, including in New Mexico. The agency cited a case in Lea County where leaks in the casing of an injection well operated by Texaco caused contamination of a farm.

> During the 1970s, 20 million gallons of salt water leaked from a Texaco disposal well in Lea County, New Mexico, into portions of a drinking water source, the Ogallala aquifer. Some of the brine made its way into a rancher's irrigation well, damaging his crop and, according to the rancher, ultimately causing the foreclosure of his farm property. On the basis of the results of a pressure test, the rancher successfully sued Texaco in 1977 for damages. Texaco subsequently made repairs to the well, and it is now operating in compliance with UIC [underground injection control] regulations. Texaco was not required to clean the aquifer, however, because, according to the Chief of New Mexico's Environment Bureau, the cost could not be economically justified.[152]

New Mexico's Governor's Office reported in 2022 that there were 1,700 abandoned oil and gas wells on private and state land.[153] (It is unclear how many are on federal land in New Mexico.) The potential for contamination through these wells is cause for concern. The state plugs about 50

Oil and gas wastewater is dumped from a truck into one of a series of unlined pits at the R360 waste disposal facility outside Hobbs, New Mexico, 2019. Photo credit: Melissa A. Troutman.

wells per year, but the governor said that the rate would significantly increase as the result of a $43.7 million infusion from the federal Interior Department provided by the federal Infrastructure Investment and Jobs Act, passed in mid-2022.

Several other types of oil and gas waste disposal could pose serious risks to New Mexicans if the waste were contaminated with PFAS. One is the disposal of oil and gas waste in earthen pits known as impoundments. New Mexico has a well-documented history of groundwater contamination due to disposal of oil and gas waste in earthen pits. From the mid-1980s to 2003, the state's Oil Conservation Division found almost 7,000 cases of soil and water contamination from oil and gas waste pits and 400 cases of groundwater contamination.[154] This evidence prompted the state to enact the "pit rule" in 2008 that prohibited those unlined pits that were most likely to cause contamination, strengthened the standards for pit liners, mandated that all pits have a permit, and banned new pits within certain distances of water resources and homes.[155] New, permanent and temporary pits, for example, were prohibited within 1,000 feet of homes, schools, or drinking water wells used by five or more families.[156] According to Earthworks, the pit rule was effective in reducing contamination: In its first two years of operation, there were no groundwater violations at pits covered by the rule. Meanwhile, oil and gas drilling expanded in the state, indicating that the rule did not hinder oil and gas extraction.[157]

However, in 2013, after opposition to the pit rule from the oil and gas industry, lawmakers passed new legislation relaxing protections. As a result, companies can now locate temporary pits containing "low chloride" fluid within 100 feet of perennial water courses, 200 feet from springs, wells or lakes, and 300 feet from homes or schools.[158] Such fluid with low chloride levels could pose risks if it were tainted with other toxics such as PFAS. Whereas the pit rule prohibited burying of waste at well sites unless the waste met more stringent health and environmental standards (e.g. benzene levels in temporary pits 50-100 feet above groundwater could not exceed 0.2 parts per million),[159] the new rule allows burying at well sites of waste under much more permissive standards (benzene levels in temporary pits 51-100 feet above groundwater cannot exceed 10 parts per million).[160] As indicated by these standards, this waste often contains dangerous contaminants including carcinogenic hydrocarbons such as benzene.[161] This report suggests that the waste could contain PFAS, too. Neither the pit rule nor the new rule mention PFAS, but by allowing for the more permissive treatment of oil and gas waste, the new rule increases the risk of contamination from waste that could contain these highly toxic and persistent pollutants.

Earthworks identified other methods of oil and gas waste disposal in New Mexico that could pose risks for PFAS contamination including taking the waste to treatment plants, recycling facilities, landfills, and “landfarms,” where contaminated soils, drill cuttings, and tank bottoms are allowed to be spread over land.[162]

c. Volatilizing, Flaring Could Pollute Air with PFAS

PFAS used in oil and gas wells could follow airborne exposure routes, according to toxicologist David Brown, former director of environmental epidemiology at the Connecticut

A poorly lit flare at Rustler Breaks SWD #6/ API #30-015-45034, a San Mateo Midstream facility in Eddy County, New Mexico, Sept. 2022. Photo credit Charlie Barrett, Earthworks.

Department of Health who has investigated health effects associated with unconventional gas drilling with the Southwest Pennsylvania Environmental Health Project. He warned that if PFAS were to enter drinking water, it could subsequently volatilize or become airborne inside homes. Brown also added another potential pathway for airborne exposure: PFAS could become airborne when gas is burned off during flaring at the wellhead or vented unburned at the wellhead.[163]

Flaring and venting are used extensively in New Mexico, suggesting that airborne PFAS through these pathways could be a risk in the state. The Howard Center for Investigative Journalism analyzed satellite data and found that between 2012 and 2020, oil and gas operators on federal land in New Mexico flared more than 138 billion cubic feet of gas,[164] enough to power more than 1.1 million homes for a year, according to a home energy consumption estimate by Popular Science magazine.[165] Gas is flared or vented unburned in emergencies and when there is insufficient pipeline capacity to bring the gas to market.[166] Insufficient pipeline capacity has been an issue in the Permian Basin in recent years when oil prices were much higher than gas prices, leaving oil and gas companies with little incentive to build pipelines to transport and sell the gas that was extracted along with the oil.[167] Soaring gas prices due to the war in Ukraine may change that equation, but it takes time to construct pipelines, and gas may not be captured if it cannot be transported to market.

In 2021, New Mexico enacted rules designed to reduce flaring and venting of gas.[168] However, some New Mexicans are skeptical that the rules can be enforced, considering that New Mexico had only 11 well inspectors as of end-2022 but 51,000 operating oil and gas wells.[169] Continued flaring and venting may provide another pathway for PFAS contamination from oil and gas wells.

Louisiana-based chemist Subra told PSR that the risk of airborne PFAS exposure might even be an issue for people living hundreds of miles from oil and gas fields.[170] Noting that gas from across the nation is delivered via pipeline to liquefied natural gas (LNG) facilities in Louisiana and Texas on the coast of the Gulf of Mexico, she proposed that residents of these communities ought to know if they are being exposed to PFAS in the gas from air emissions related to transforming the gas into a liquid for export. Bolstering Subra's concern, Reuters reported that in 2020, an LNG export facility in Corpus Christi, Texas operated by Cheniere Energy, Inc., exceeded permitted limits for air emissions in 293 instances. At least some of the emissions were volatile organic compounds from chemicals removed from the natural gas during the liquefaction process.[171] Reuters did not report that PFAS was released, but it is unclear whether anyone monitored for it. Some of the gas to be exported as LNG that could contain PFAS may arrive at the Gulf Coast from New Mexico. The Energy Information Administration reported in 2022 that three new pipelines will allow gas producers in the Permian basin to reach LNG export facilities on Texas's Gulf Coast.[172]

CH. 5 HEALTH STUDIES LINK OIL AND GAS OPERATIONS TO ILLNESS

a. Oil, Gas Well Proximity Associated with Disease

Peer-reviewed studies of people living near oil and gas operations have found that proximity to active well sites correlates with a variety of diseases and other health effects. While studies are lacking on health effects in New Mexico, a 2021 study of more than three million pregnant women in Texas showed that living within one kilometer of an active oil or gas well increased the odds of gestational hypertension (high blood pressure) and eclampsia[173] (a pregnancy-related high blood pressure disorder that can induce seizures or coma).[174] A 2020 study of pregnant women living in the Eagle Ford Shale area of South Texas found that exposure to a high number of nightly flaring events was associated with a 50 percent increase in the risk of preterm birth.[175] A 2020 study in Texas documented a link between natural gas drilling and production from both conventional and unconventional wells and frequency of hospitalization for childhood asthma.[176] Several studies conducted in Colorado, another major producer of oil and gas, also found associations between proximity to oil and gas operations and health effects, including congenital heart defects in newborns[177] and cancer diagnoses among Coloradans from birth to 24 years old.[178]

PSR has collaborated with Concerned Health Professionals of New York to compile and summarize the substantial and growing number of scientific studies that have found serious health effects associated with oil and gas operations. In the eighth edition (2022) of our report, we wrote,

> Public health problems associated with fracking include prenatal harm, respiratory impacts, cancer, heart disease, mental health problems, and premature death…. Poor birth outcomes have been linked to fracking activities in multiple studies in multiple locations using a variety of methods. Studies of mothers living near oil and gas extraction operations consistently find impaired infant health, especially elevated risks for low birth weight and preterm birth. As we go to press, a new study in Pennsylvania finds "consistent and robust evidence that drilling shale gas wells negatively impacts both drinking water and quality of infant health."[179]

Low birthweight is a leading contributor to infant death in the United States.[180]

Many residents living near oil and gas operations have reported serious health concerns while expressing frustration over the secrecy surrounding chemicals used by the oil and gas industry.[181] In 2020, Pennsylvania's Attorney General issued a report based on a criminal grand jury investigation of oil and gas drilling pollution in the Keystone State. Drilling for gas in shale formations has surged in that state over the past 15 years,[182] vaulting it into the number two spot among gas-producing states (Texas is number one)[183] and bringing many more Pennsylvanians into contact with gas drilling and its impacts. Based on testimony from over 70 households, the attorney general compiled evidence of serious health impacts, finding that

> Many of those living in close proximity to a well pad began to become chronically, and inexplicably, sick. Pets died; farm animals that lived outside started miscarrying, or giving birth to deformed offspring. But the worst was the children, who were most susceptible to the effects. Families went to their doctors for answers, but the doctors didn't know what to do. The unconventional oil and gas companies would not even identify the chemicals they were using, so that they could be studied; the companies said the compounds were "trade secrets" and "proprietary information." The absence of information created roadblocks to effective medical treatment. One family was told that doctors would discuss their hypotheses, but only if the information never left the room.[184]

b. Studies Needed on PFAS

PSR is not aware of published studies that have analyzed well sites for PFAS or that have analyzed health effects related to potential use of PFAS at well sites. This lack of testing is not surprising; there were few if any grounds to

Angel Peak Scenic Area, Farmington, New Mexico, May 2012. New Mexico's natural beauty is well worth protecting. Photo credit: Judy Gallagher, https://creativecommons.org/licenses/by/4.0/.

test for PFAS in connection with oil and gas operations prior to July 2021, when PSR first publicized the probable use of these chemicals in oil and gas extraction. Now that we know PFAS have been used in oil and gas operations for years, scientists should determine whether there are connections between this use and health effects, for PFAS chemicals individually and as a compounding factor in conjunction with exposure to other fracking chemicals.

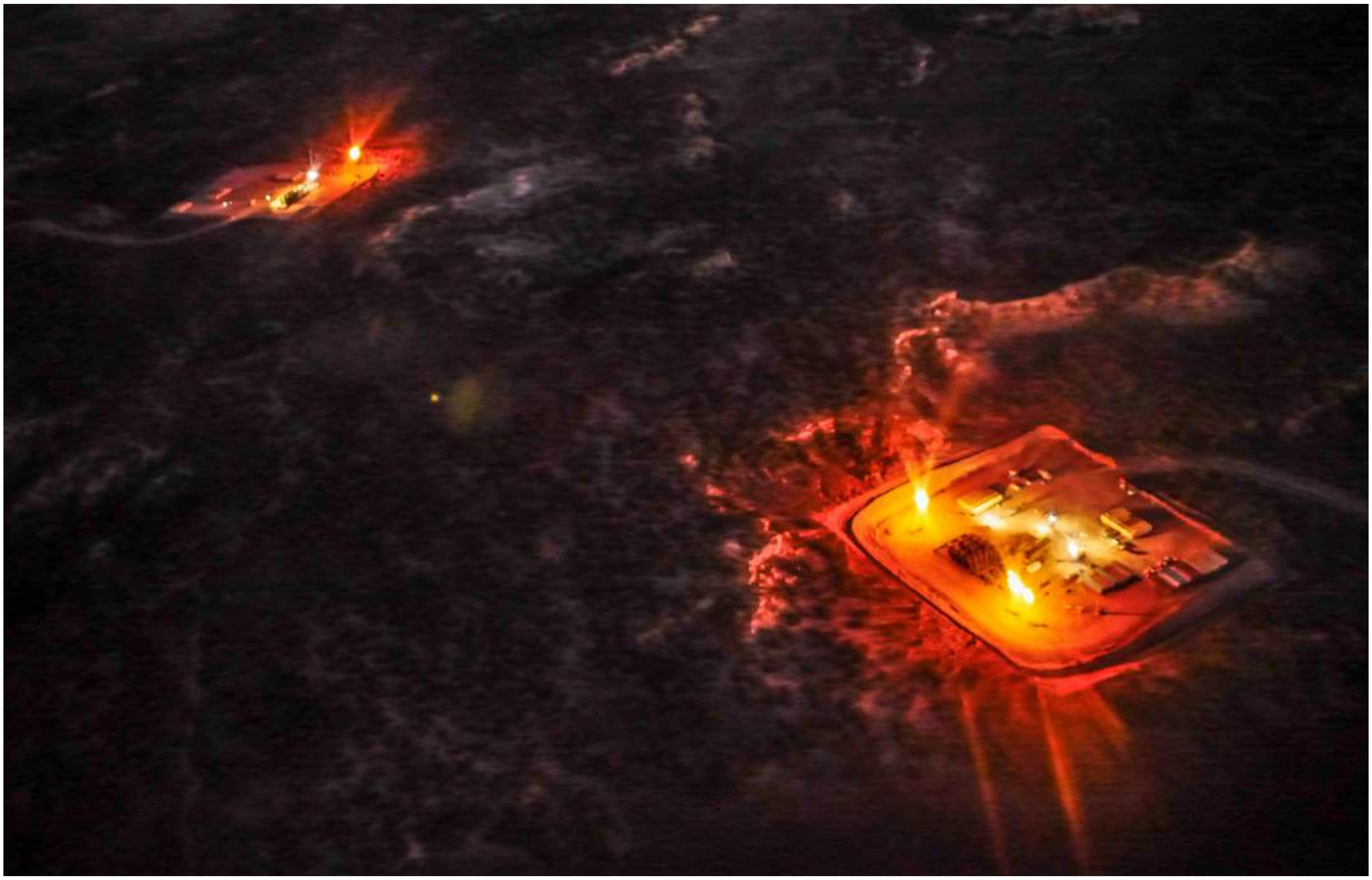
CH. 6 OIL & GAS-RELATED CHEMICAL EXPOSURE AS AN ENVIRONMENTAL JUSTICE ISSUE

a. Disproportionate Impacts on Environmental Justice Communities

“Fenceline” communities – people living close to oil and gas operations – often bear a disproportionate risk of exposure to toxic chemicals and thus may be particularly at risk from PFAS used in oil and gas extraction. Although drilling and fracking take place in the majority of U.S. states, not everyone shares in the risks equally. Rather, oil and gas infrastructure and associated chemicals are frequently located in or adjacent to lower-income, underserved, and marginalized communities, notably Black, Indigenous, and other communities of color.

In 2021, researchers used satellite observations and census data to show that 83 percent of the flaring from unconventional oil and gas wells in the contiguous United States between March 2012 and February 2020 took place in three basins: the Permian Basin in New Mexico and Texas, the Williston Basin in North Dakota, and the Western Gulf Basin in southern Texas and Louisiana. They estimated that over half a million people in these basins lived within three miles of a flare, with 39 percent of them living near more than 100 flares each night. The researchers also reported that in these regions, Black, Indigenous, and people of color were disproportionately exposed to flaring.[185]

Other studies have also found disproportionate impacts on people of color. A 2020 study found that compared to white residents, Hispanic residents living in the Eagle Ford shale region of Texas were disproportionately exposed to



Nighttime flaring, just north of Chaco Culture National Historical Park near Nageezi, New Mexico, Oct. 2014. Photo credit: Dom Smith, EcoFlight.

flaring from unconventional oil and gas wells, even though they were less likely than white residents to live near unconventional oil and gas wells.[186] In 2016, a public health research team showed that in the Eagle Ford shale region, disposal wells for fracking wastewater were more than twice as common in areas where residents were more than 80 percent people of color than in majority-white communities. They also found that disposal wells were disproportionately located in areas with high rates of poverty, but even in these areas, the association with race was predominant. "Adjusting for both poverty and rurality," the researchers wrote, "we still found that as the proportion of people of color in the census block group increased, so did the presence of disposal wells." Since 2007, they reported, Texas had permitted more than 1,000 waste disposal wells in the Eagle Ford Shale region, where groundwater is the primary source of drinking water.[187]

A 2019 analysis conducted in Colorado, Oklahoma, Pennsylvania, and Texas found strong evidence that African Americans disproportionately lived near fracking wells in Texas and Oklahoma, while Hispanics disproportionately lived near fracking wells in Texas and urban Colorado. "The question, who bears the costs of unconventional natural gas drilling, is of great relevance not only for the U.S., but worldwide," the researchers wrote.

b. Navajo Survey Shows Health Impacts

All chapters of the Navajo Nation in New Mexico were identified in 2020 as "environmental justice" communities by the United States Bureau of Land Management.[188] In 2021, the Counselor Chapter of the Navajo Nation in New Mexico conducted a health and cultural survey regarding oil drilling operations in the Counselor, Torreon, and Ojo Encino chapters that identified health risks and distrust of regulators and oil companies. The chapter conducted its health survey under the guidance of the Southwest Pennsylvania Environmental Health project, which had conducted similar surveys in other communities with oil and gas drilling. Among other things, the chapter measured the levels of fine particulate matter ($PM_{2.5}$) through air monitors near people's

Table 9. Wells on NM Federal, State, and Tribal Land Fracked with PFAS and Possible PFAS, 2013-2022

Type of fracking chemical injected	No. Wells in state	Total Mass in state (lbs.)	No. Wells on Federal Land	Total Mass Federal Land (lbs.)	No. Wells on State Land	Total Mass State Land (lbs.)	No. Wells on Tribal Land	Total Mass Tribal Land (lbs.)
	9066	--	4468	--	2350	--	192	--
Trade Secret chemicals	8293	243,000,000	4072	115,000,000	2153	54,600,000	186	2,040,000
Trade Secret surfactants	3681	19,300,000	1813	10,900,000	954	4,740,000	86	230,000
Fluoro-surfactants	24	965	12	790	10	164	0	0.0
65545-80-4	34	6,400	8	1,370	17	3,060	0	0.0
PTFE	227	2,610	113	1,650	53	552	3	data not available

This table shows the number of oil and gas wells in New Mexico -- statewide, on federal land, on state-owned land, and on tribal land – that oil and gas companies fracked between 2013 and 2022 with at least one trade secret chemical, at least one trade secret surfactant, at least one fluorosurfactant, fluoroalkyl alcohol substituted polyethylene glycol (CAS Number 65545-80-4, a known PFAS), or PTFE (a known PFAS). The total weight figures reflect the sum of all records for which PSR has enough information to calculate a chemical's weight. For a detailed explanation of data sources, see the Appendix.

homes on the side of the home nearest to the closest oil wells. The readings showed the $PM_{2.5}$ levels, generally recorded between peak releases, were significantly higher at six of eight measuring sites compared to median levels in other non-Navajo communities with oil and gas operations. Residents living near a source of such air pollution are at greater risk for contracting or intensifying respiratory or cardiovascular diseases.[189] In a survey of health symptoms of 80 residents of the Counselor Chapter, more than 60 percent reported 11 symptoms during the year after drilling began near their homes, including sore throat, cough, and sinus problems. This number of reported health symptoms was greater than the number reported by respondents living near oil and gas wells in other communities in the U.S.[190] Separately, the Chapter conducted a cultural survey regarding the effects of oil drilling, collecting data from 136 randomly selected adults in the three chapters. Among other findings, 104 respondents strongly agreed with the statement, "Our local leaders have spoken out against drilling and no one at the tribal, state or federal level, including BLM and BIA, has listened." One hundred and seventeen strongly agreed with the statement, "The oil companies have no respect for land, people & life."[191]

PSR found that about 97 percent of the wells in New Mexico drilled on tribal land for which oil and gas companies disclosed the use of fracking chemicals were injected with at least one trade secret fracking chemical. This percentage was a bit higher than for wells drilled statewide (about 91 percent). But the total number of wells drilled on tribal land was much smaller, so it is unclear whether this difference was statistically significant.

Where a pattern of risks affects people of color and/or lower-income people disproportionately, oil and gas production methods should be viewed and addressed as an Environmental Justice issue. So too should any oil and gas-related exposure to PFAS.

CH. 7

POLICY CAN HELP PROTECT NEW MEXICANS FROM PFAS IN OIL & GAS OPERATIONS

a. Modest Federal Protections from PFAS Pollution

Governments at all levels will have to do more to protect the public from PFAS, in large part because EPA has taken only modest steps to do so, while Congress and the executive branch have exempted the oil and gas industry from major provisions of multiple federal environmental laws. For example, oil and gas waste is exempted from the hazardous waste rules that require cradle-to-grave tracking and safe handling of hazardous substances under the Resource Conservation and Recovery Act. These exemptions increase the burden on state governments to address any PFAS pollution associated with oil and gas extraction.[192]

EPA has taken some steps to protect the public from dangerous PFAS. In 2005, EPA reached a then-record $16.5 million settlement with chemical manufacturer Dupont after accusing the company of violating the federal Toxic Substances Control Act (TSCA) by failing to disclose information about PFOA's toxicity and presence in the environment.[193] In 2006, EPA invited Dupont, 3M and six other companies to join a "stewardship" program in which the companies promised to achieve a 95 percent reduction of emissions of PFOA and related chemicals by 2010, compared to a year 2000 baseline. The agreement also required the companies to eliminate such emissions and use of these chemicals by 2015.[194] In 2022, EPA said on its website that the companies reported that they had accomplished those goals, either by exiting the PFAS industry or by transitioning to alternative chemicals.[195] EPA reported in 2022 that the manufacture and use of at least one PFAS, PFOA, had been phased out in the U.S., and that no chemical company had reported making PFOS in the U.S. since 2002. EPA noted that existing stocks of PFOA might still be used, and imported products may contain some PFOA.[196] A 2020 scientific article reported that PFOA was still used in Asia.[197] EPA stated that limited ongoing uses of PFOS remain.[198] Since the announcement of its PFAS stewardship program in 2006, EPA has allowed nearly unlimited use of closely related "replacement" chemicals in dozens of industries.[199] In response, in 2015 a group of more than 200 scientists raised health and environmental concerns that the new PFAS designed to replace PFOA and PFOS may not be safer for health or the environment.[200]

In October 2021, EPA announced a "strategic roadmap" for regulating PFAS. This plan encompasses a goal of setting federal drinking water standards for several PFAS chemicals by 2023, as well as commitments to "use all available regulatory and permitting authorities to limit emissions and discharges from industrial facilities" and "hold polluters accountable."[201] The plan does not, however, include an examination of PFAS use in the oil and gas industry. (Later that month, 15 members of the U.S. House of Representatives asked EPA to examine this topic.[202] The month before, PSR asked EPA to collect data on PFAS use in oil and gas extraction, utilizing its authority under TSCA.[203] As previously stated, in June 2022, EPA announced new health advisory levels for several types of PFAS; unfortunately, these standards are advisory and not legally enforceable.[204] In August 2022, EPA proposed designating PFOA and PFOS as hazardous under Superfund.[205] This designation would enable affected parties to more easily hold oil and gas companies accountable for cleanup costs if PFOA and PFOS were found at oil and gas sites because under Superfund, liability does not require negligence, and any potentially responsible party (PRP) can be held liable for cleanup of an entire site when it is difficult to distinguish contributions to pollution among several parties. As EPA writes about Superfund, "[i]f a PRP sent some amount of the hazardous waste found at the site, that party is liable."[206] Finally as previously stated, in March 2023, EPA announced a plan to regulate six types of PFAS in drinking water.

In acting belatedly to regulate at least some types of PFAS in drinking water, EPA is following the lead of several states. As of 2023 nine states, including at least several with contaminated military sites, had developed enforceable standards for concentrations of several types of PFAS in drinking water.[207] One of those to act is Michigan, which set standards in 2020 for limiting PFAS in drinking water and for removing PFAS from groundwater. The standards apply to PFOA and six other forms of PFAS. Michigan's maximum allowable level is no more than eight parts per trillion for PFOA,[208] a standard that is one of the lowest among states but is now much more permissive than EPA's interim health advisory level. Even Michigan's standard, however, shows how toxic PFAS can be. By extrapolation, Michigan's standard suggests that one measuring cup of PFOA could contaminate almost eight billion gallons of water – the amount of water needed to fill almost 12,000 Olympic-sized swimming pools at about 660,000 gallons per pool.[209]

b. New Mexico Disclosure Rules: In Need of Sweeping Reform

In New Mexico, multiple reforms are needed to protect the public from the use of PFAS in oil and gas operations, including changing the state's chemical disclosure rules to lift the veil of secrecy that oil and gas companies have used to conceal the use of potentially dangerous chemicals including, perhaps, PFAS. One such change should be tighter limits on the use of trade secret provisions.

Oil and gas companies have argued that chemical trade secrets are necessary to protect their intellectual property from competitors. However, this interest does not have to mean a complete withholding of information on chemical identities from scientists, regulators, and the public. In 2015, California, a major oil-producing state,[210] began requiring full disclosure of chemicals used for well stimulation, including fracking. The policy did away with trade secret exemptions for the individual chemicals used in fracking products.[211] In June 2022, Colorado, a major producer of oil and gas,[212] followed in California's footsteps but extended the disclosure requirements to all chemicals used in oil and gas wells, not just fracking or stimulation chemicals.[213]

The methodology utilized in California and Colorado is consistent with a recommendation issued in 2014 by an advisory panel to the U.S. Department of Energy: that companies reveal the fracking chemicals injected into each well, providing that information in a list in which the chemicals are disassociated from the trade name of the commercial products they are part of.[214] This form of disclosure enables the public to know all the chemicals used in fracking without disclosing to rival chemical manufacturers the exact components of proprietary formulas.[215] In a similar way, food producers keep recipes secret while disclosing individual ingredients, enabling the public to know the contents of food products but making it difficult for rival producers to recreate valuable food brands. In addition, California has a process under which state regulators review secrecy requests from chemical companies to determine whether the information must be kept proprietary.[216] Health and safety data related to fracking fluids are not allowed to be hidden from public view under California law.[217] California also requires disclosure of fracking chemicals before fracking begins,[218] as do West Virginia[219] and Wyoming.[220]

New Mexico should also ensure that full chemical disclosure is required from all the companies in the chemical supply chain. Currently, New Mexico rules require chemical disclosure from the well operator.[221] Chemical manufacturers, however, are exempted from this reporting, despite being the only entity that always knows the precise contents of the chemicals they produce. Not only does New Mexico omit chemical manufacturers from disclosure requirements and allow them to claim trade secrets; it also limits their responsibility by providing that the Division of Oil and Gas "does not require the reporting of information beyond the material safety data sheet data as described in 29 C.F.R. 1910.1200." This provision means that disclosure is limited to what is required on material safety data sheets (now called safety data sheets) on which chemical manufacturers list information about their chemicals to protect workers. Well operators are not responsible for compiling chemical information from manufacturers that is not disclosed on the sheets.[222] As several Harvard researchers reported in 2013, manufacturers can legally omit chemical information from the sheets. For example, if a chemical has not been tested and found to be hazardous, it does not need to be disclosed, even if tests would show that it is hazardous.[223] Therefore, the manufacturers could effectively withhold this information from public disclosure with or without trade secret protection.

Flaring near Chaco Culture National Historic Park, Dec. 2014. Photo credit: Jane Pargiter, EcoFlight.

Evidence suggests that chemical manufacturers do not always tell companies farther down the supply chain the full contents of the chemical products they are using; rather, they provide these companies with vague descriptions, generic chemical family names or, as the Harvard researchers suggested, Material Safety Data Sheets with an incomplete list of chemicals.[224] In such cases, the end users may legitimately be unable to disclose all the identities of chemicals – including PFAS – used at a particular well, whether under trade secret protection or not. They simply would not have the information. Requiring disclosure of oil and gas chemicals by chemical manufacturers would avoid this problem. Colorado took this step in its June 2022 legislation.[225]

These reasonable and feasible reforms are valuable steps to protect the health of people who may be exposed to PFAS and other dangerous oil and gas chemicals, be they industry workers, residents living near well sites, or first responders called to the scene of an accident. They can improve health and potentially save lives. Additional steps to reduce the harms caused by oil and gas extraction are outlined in the following section, including a ban on the use of PFAS in oil and gas operations, an action that Colorado took in 2022.[226] Among the evidence supporting the feasibility of this measure is a peer-reviewed analysis published in 2021 showing that many PFAS are immediately replaceable with less-persistent and less-toxic substances, including for use in the oil and gas industry.[227]

c. New Mexico Hazardous Waste Rules Also in Need of Reform

New Mexico's state government has recognized the dangers of PFAS but, in doing so, has illuminated another gap in state rules that should be closed to protect the public from PFAS use in oil and gas operations. In 2021, Governor Michelle Lujan Grisham petitioned EPA to list the class of chemicals known as PFAS as hazardous under Subtitle C of the federal Resource Conservation and Recovery Act (RCRA) or, alternatively, "list individual PFAS chemicals under RCRA known to have harmful effects to humans and the environment."[228] Subtitle C of RCRA is our nation's law that requires safe management of hazardous waste from "cradle-to-grave."[229] Gov. Luhan Grisham emphasized how important this policy change would be for New Mexico, writing

> I implore EPA to do what is immediately necessary to protect the people and environment of the United States from the real and potentially devastating effects of exposure to PFAS....Without a uniform regulatory process addressing PFAS from manufacture to disposal, states like New Mexico will be left attempting to use a patchwork of statutory and regulatory authorities that may or may not provide enough oversight...[230]

EPA administrator Michael Regan replied later in 2021 that the agency would initiate a rulemaking process to declare four types of PFAS to be hazardous under RCRA: PFOA, PFOS, PFBS, and GenX. He also said that EPA would initiate a rulemaking to "clarify that emerging contaminants such as PFAS can be addressed through RCRA corrective action."[231]

Yet under both the federal RCRA[232] and the state's implementation of the federal law,[233] oil and gas wastes are exempt from hazardous waste requirements. This exemption likely means that even if EPA acted on the governor's petition and declared PFAS hazardous, oil and gas wastes containing PFAS would not be subject to hazardous waste protections. New Mexico could act to avoid this problem and regulate oil and gas waste as hazardous by following the example of New York State. In 2020, New York enacted legislation to designate oil and gas waste as hazardous.[234] State Senator Rachel May, one of the bill's sponsors, said in a statement,

> Wastewater from fracking can contain carcinogenic compounds and naturally occurring radioactive materials. The regulatory loophole that allowed waste from fracking and crude oil processing to be treated as standard industrial waste means it enters local sewage treatment facilities, sometimes with radiation levels hundreds of times the safe limit, it then flows directly back into our waterways – the source of drinking water for thousands of New Yorkers.[235]

May issued her statement before it was widely known that PFAS was used in oil and gas operations, but considering the oil and gas industry's record of using PFAS, these chemicals could be present in oil and gas wastes whether in New York, New Mexico, or other states. Continuing to exempt oil and gas wastes from hazardous waste treatment means that PFAS in these wastes would likely be exempt, too, with potentially serious consequences for New Mexicans.

RECOMMENDATIONS

In light of the findings shared in this report, PSR recommends the following:

- **Halt PFAS use in oil and gas extraction.** New Mexico should follow the lead of Colorado, a major oil- and gas-producing state that in June 2022 passed legislation banning the use of PFAS in oil and gas wells. Furthermore, New Mexico and the U.S. Environmental Protection Agency (EPA) should prohibit PFAS from being used, manufactured, or imported for oil and gas extraction. Many PFAS are replaceable with less-persistent and less-toxic alternatives.

- **Expand public disclosure.** New Mexico should greatly expand its requirements for public disclosure of oil and gas chemicals. TThe state could again follow the example offered by Colorado by requiring disclosure of all individual chemicals used in oil and gas wells, without exceptions for trade secrets, while still protecting chemical product formulas. New Mexico should also require disclosure on the part of chemical manufacturers and require chemical disclosure prior to permitting, as have California, West Virginia, and Wyoming.

- **Increase testing and tracking.** New Mexico and/or the U.S. EPA should determine where PFAS have been used in oil and gas operations in the state and where related wastes have been deposited. They should test nearby residents, water, soil, flora, and fauna for PFAS, both for the particular type(s) of PFAS used and for organic fluorine to detect the presence of other PFAS. and/or their breakdown products. Testing equipment should be used that is sensitive enough to detect PFAS at a level of single-digit parts per trillion or lower.

- **Require funding and cleanup**. Oil and gas and chemical firms should be required to fund environmental testing for PFAS in their areas of operation, and should PFAS be found, be required to fund cleanup. If water cleanup is impossible, companies responsible for the use of PFAS should pay for alternative sources of water for household and agricultural uses, as needed.

- **Remove New Mexico's oil and gas hazardous waste exemption.** New Mexico exempts oil and gas industry wastes from state hazardous waste rules. New Mexico should follow New York's lead and remove its state-level hazardous waste exemption for the oil and gas industry.

- **Reform New Mexico's regulations for oil and gas production wells and underground injection disposal wells.** The state should prohibit production wells and underground wastewater disposal wells close to underground sources of drinking water, homes, health care facilities and schools, require groundwater monitoring for contaminants near the wells, and for disposal wells, require full public disclosure of chemicals in the wastewater.

- **Transition to renewable energy and better regulation.** Given the use of highly toxic chemicals in oil and gas extraction, including but not limited to PFAS, as well as climate impacts of oil and gas extraction and use, New Mexico should transition away from oil and gas production and move toward renewable energy and efficiency while providing economic support for displaced oil and gas workers. As long as drilling and fracking continue, the state should better regulate these practices so that New Mexicans are not exposed to toxic substances and should empower local governments also to regulate the industry. When doubt exists as to the existence or danger of contamination, the rule of thumb should be, "First, do no harm."

APPENDIX

Data Sources for PFAS Used in New Mexico's Oil and Gas Wells

To identify where PFAS were used at oil and gas wells in New Mexico, PSR analyzed data from the state Oil Conservation Division that is part of the Energy, Minerals and Natural Resources Department. These data, based on reports from oil and gas well operators, show well-by-well which fracking chemicals were used.236 These data date from January 1, 2013 to early 2018, likely because a change in state rules in September 2017 required reporting to the FracFocus database rather than to the state.[237] PSR also relied on the well-by-well reports of fracking chemicals recorded in FracFocus, a database for the oil and gas industry[238] maintained by the Groundwater Protection Council,[239] a nonprofit comprised of regulators from state agencies. The dates of these records extend from January 1, 2013 to September 29, 2022. PSR consulted the open-source version of FracFocus, Open-FF,[240] which is more accurate and informative than the original version of FracFocus.[241]

Under current New Mexico law, operators must disclose the fracking chemicals used in each well to the FracFocus database using the "current edition of the hydraulic fluid product component information form published by FracFocus." Disclosure must occur within 45 days after hydraulic fracturing treatment.[242] Based on the disclosure forms available on FracFocus' website, operators must list, among other things, each individual chemical injected into the well and each chemical's CAS number, if available.[243] New Mexico's prior fracking chemical disclosure rules required disclosure of similar information.[244] There are, however, significant exceptions to disclosure requirements under New Mexico's rules, including an exception for chemicals designated a trade secret[245] that are discussed in Chapter 3 and Chapter 7.

Table 6. Oil and Gas Companies that Fracked Wells in New Mexico Using Trade Secret Chemicals and Trade Secret Surfactants, 2013-2022.

Operator	Number of wells injected with trade secret chemicals	Number of wells injected with trade secret surfactants
EOG Resources, Inc.	1177	214
COG Operating LLC	844	438
Devon Energy Production Company L. P.	586	358
Mewbourne Oil Company	575	116
Occidental Oil and Gas	498	141
XTO Energy/ExxonMobil	442	203
Apache Corporation	439	386
Cimarex Energy Co.	336	186
Matador Production Company	288	63
Chevron USA Inc.	264	189
Hilcorp Energy Company	203	0
ConocoPhillips Company/Burlington Resources	161	112
WPX Energy	148	21
Kaiser-Francis Oil Company	131	67
Lime Rock Resources Ii-A, L.P.	129	91
BTA Oil Producers LLC	121	27
Marathon Oil	108	3
Mack Energy Corp	105	76
RKI Exploration & Production, LLC	100	55
Burnett Oil Co., Inc.	99	47
BOPCO, L.P.	86	64
Yates Petroleum Corporation	84	71
LRE Operating, LLC	82	64
Encana Oil & Gas (USA) Inc.	76	40
Tap Rock Resources	73	1
Centennial Resource Production, LLC	70	10
DJR Operating, LLC	70	63
Energen Resources Corporation	68	20
Advance Energy Partners Hat Mesa LLC	53	22
Murchison Oil & Gas Inc	47	44
Dugan Production Corp.	46	27
Novo Oil & Gas Texas, LLC	44	39
Logos Operating, LLC	42	21
Franklin Mountain Energy	39	0
Enduring Resources LLC	36	26
Vanguard Permian LLC	34	14
BP America Production Company	33	16
OXY USA WTP Limited Partnership	33	23

Table 6. Oil and Gas Companies that Fracked Wells in New Mexico Using Trade Secret Chemicals and Trade Secret Surfactants, 2013-2022.

Operator	Number of wells injected with trade secret chemicals	Number of wells injected with trade secret surfactants
Titus Oil & Gas Production, LLC	30	16
Caza Operating, LLC	23	13
Endurance Resources LLC	23	22
Chisholm Energy Operating, LLC	21	2
Legacy Reserves Operating LP	21	10
Ameredev Operations LLC	20	5
EnerVest, Ltd.	20	19
SM Energy	18	2
Colgate Operating, LLC	16	1
Gmt Exploration Company LLC	15	14
BreitBurn Operating LP	14	8
Burlington Resources Oil & Gas Company LP	14	14
Nearburg Producing Co	14	14
Longfellow Energy, LP	13	9
Redwood Operating LLC	13	4
Percussion Petroleum LLC	12	10
Steward Energy II, LLC	12	7
Fasken Oil & Ranch Ltd	11	10
Nadel and Gussman Permian, LLC	11	10
Read & Stevens, Inc.	10	10
Whiting Petroleum	10	3
Linn Operating, Inc.	9	2
Cross Timbers Energy, LLC	8	6
Pride Energy Company	8	8
Strata Production Co.	8	6
Chesapeake Operating, Inc.	7	7
Elm Ridge Exploration Company LLC	7	0
Forty Acres Energy LLC	7	6
Legend Natural Gas Iii Limited Partnership	7	1
McElvain Energy, Inc.	7	5
Regeneration Energy, Corp	7	6
Alamo Permian Resources, LLC	6	5
Manzano LLC	5	4
V-F Petroleum Inc	5	3
Atlas Energy, L.P.	4	0
Avant Operating, LLC	4	0
Capstone Natural Resources, LLC	4	4
Lynx Petroleum Consultants Inc	4	4

Table 6. Oil and Gas Companies that Fracked Wells in New Mexico Using Trade Secret Chemicals and Trade Secret Surfactants, 2013-2022.

Operator	Number of wells injected with trade secret chemicals	Number of wells injected with trade secret surfactants
Marshall & Winston Inc	4	4
Premier Oil & Gas Inc	4	4
Rockcliff Energy Operating	4	0
Special Energy Corporation	4	4
Hadaway Consulting and Engineering, LLC	3	3
Nemo Fund I, LLC	3	3
Stephens & Johnson Operating Co.	3	1
Sundown Energy LP	3	0
Catena Resources Operating, LLC	2	2
Foundation Energy Management, LLC	2	2
IACX Production	2	2
ICA Energy Operating LLC	2	0
Koch Exploration Company, LLC	2	2
Maverick Operating, LLC	2	2
Memorial Resource Development LLC	2	0
OneEnergy Partners Operating, LLC	2	0
Quantum Resources Management, LLC	2	2
Sg Interests I Ltd	2	2
SIMCOE LLC	2	0
Texland Petroleum, LP	2	2
Thompson Engr & Prod Corp	2	1
Amtex Energy Inc.	1	0
BAM Permian Operating, LLC	1	1
BC Operating, Inc.	1	1
Boaz Energy, LLC.	1	1
Chuza Oil Company	1	1
Clayton Williams Energy Inc.	1	0
CML Exploration, LLC	1	1
Cobra Oil & Gas Corporation	1	1
D J Simmons Inc	1	1
DGP Energy	1	0
Forge Energy, LLC	1	1
Harvey E Yates Co	1	1
HEXP Operating, LLC	1	1
Hunt Cimarron Limited Partnership	1	1
Huntington Energy, LLC	1	0
ImPetro Operating LLC	1	1
Mammoth Exploration, LLC	1	1

Table 6. Oil and Gas Companies that Fracked Wells in New Mexico Using Trade Secret Chemicals and Trade Secret Surfactants, 2013-2022.

Operator	Number of wells injected with trade secret chemicals	Number of wells injected with trade secret surfactants
Mar Oil & Gas Corp.	1	0
Merit Energy Company	1	1
Merrion Oil & Gas Corp	1	1
Ridgeway Arizona Oil Corp.	1	0
Robert L. Bayless, Producer LLC	1	1
Running Horse Production Company	1	0
San Juan Resources, Inc.	1	0
Seely Oil Co	1	1
Tacitus LLC	1	0
Western Refining Southwest, Inc.	1	0

This table shows the oil and gas companies that fracked oil and gas wells in New Mexico with trade secret chemicals and trade secret surfactants between January 1, 2013 and September 29, 2022. The wells injected with trade secret surfactants are a subset of the wells injected with trade secret chemicals.

*ND = No data available.

New Mexico must strengthen its protections from PFAS and other pollution related to oil and gas extraction to safeguard its land and people. View from Deep Access Cave, Carlsbad Caverns National Park, Sept. 2020. Photo credit: Dan Pawlak, National Park Service.

ENDNOTES

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methodology, we will use as an example the figures from XTO Energy/ExxonMobil's well number 35-019-26303 fractured in Carter County, Oklahoma in 2019. We estimated the total mass of the hydraulic fracking fluid used in each well in pounds by multiplying the gallons of water listed as being used as the base fluid for the fracking fluid (223,650 in this case) by 8.33, the number of pounds in a gallon of water as listed in a table of the weights of various solvents published by the U.S. Environmental Protection Agency. See U.S. Environmental Protection Agency. Conversion from Gallons to Pounds of Common Solvents. Accessed Jan. 12, 2022, at https://www.epa.gov/p2/pollution-prevention-tools-and-calculators. That quantity of water in the XTO Energy/ExxonMobil example weighs approximately 1,863,005 pounds. We then calculated the total mass of the fracking fluid by multiplying the mass of the water in pounds by 100 and dividing that product by the listed maximum percent concentration of water in the fracking fluid (78.31797). The estimated total maximum mass of the fracking fluid in the example is 2,378,770 pounds. Next, we multiplied the listed maximum concentration in percent by mass of the potential PFAS chemical in the fracking fluid (0.00074) by the total estimated mass of the fluid. The result was an estimated maximum of 17.6 pounds of potential PFAS used to fracture the well. PSR included in our analysis of trade secret chemicals those chemicals in Open-FF whose specific identities were explicitly labeled "proprietary," "trade secret," or "confidential business information" in place of a CAS number. PSR did not include as trade secrets additional unidentified chemicals for which the CAS number in Open-FF is blank.

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methodology, we will use as an example the figures from XTO Energy/ExxonMobil's well number 35-019-26303 fractured in Carter County, Oklahoma in 2019. We estimated the total mass of the fracking fluid used in each well in pounds by multiplying the gallons of water listed as being used as the base fluid for the fracking fluid (223,650 in this case) by 8.33, the number of pounds in a gallon of water as listed in a table of the weights of various solvents published by the U.S. Environmental Protection Agency. See U.S. Environmental Protection Agency. Conversion from Gallons to Pounds of Common Solvents. Accessed Sept. 9, 2022, at https://www.epa.gov/p2/pollution-prevention-tools-and-calculators. That quantity of water in the XTO Energy/ExxonMobil example weighs approximately 1,863,005 pounds. We then calculated the total mass of the fracking fluid by multiplying the mass of the water in pounds by 100 and dividing that product by the listed maximum percent concentration.

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