AIR LIQUIDE
SPECIALTY GASES

What's really inside?

Everything you wanted to know about EPA's Protocol Gas Verification Program

EPA's PGVP Initiative

Original intent behind the creation of the PGVP

Since the implementation of "cap and trade" environmental controls for Acid Rain in 1996, SO_2 emissions in the United States have been reduced by more than 50%. The heart of this very successful, market-driven control concept is the widespread deployment of continuous emissions monitoring systems (CEMS), the overwhelming majority of which are calibrated using EPA protocol gases. It is the EPA protocol gas that sets the reported emission value for a source and ultimately determines the cost for emissions allowances for that source. Because of the tremendous economic leverage of these measurements, the accuracy of EPA protocol gases must be unquestioned.

However, persistent feedback from source owners claimed that the stability and accuracy of EPA protocol gases was not consistent across the specialty gas industry, that there was a need for improvement. Indeed, intermittent blind audits conducted by the EPA from 1996 to 2010 seemed to support these claims, but EPA funding was insufficient to support a protocol gas audit every year. Therefore, the EPA initiated the PGVP, which guarantees annual mixture verifications performed by the National Institute of Standards and Technology (NIST), and to be funded by the producers of EPA protocol gases. EPA's goals with this new program are to ensure that EPA protocol gases meet the accuracy requirements of 40 CFR Part 75, and to encourage industry-wide improvement in quality. Although participation by specialty gas vendors is voluntary, Part 75 sources are required to obtain EPA protocol gases from a production site that participates in the PGVP.

Annual audit results from NIST are published on the EPA website and are publicly accessible. Though technically not a true quality program, the PGVP results provide a snapshot of vendor performance to purchasers of EPA protocol gases and thereby assist them in making purchasing decisions. Likewise, the publicized results indirectly provide an incentive for those gas vendors who performed poorly in audits to make improvements to their products.

Air Liquide protests implementation of the PGVP in its original form

As a leading supplier of EPA protocol gases with a consistent record of outstanding performance in EPA audits, Air Liquide had serious concerns with the original design of the PGVP that would ultimately have a negative impact on both customers and other gas suppliers. Our concerns included:

> Technical Content – The proposed small audit sample size (no more than four cylinders annually) creates risk that gas customers may draw misleading conclusions about vendor performance and overall gas quality.

> Program Fees – The potential for high program costs, coupled with inequitable "per facility" program fee assessment, would be passed along to customers, raising the cost of EPA protocol gases unnecessarily.

> Quality Improvement – Vendor gas quality would be passively judged by audit results publication; the PGVP would not necessarily force any appreciable vendor improvement.

> Program Administration – The Institute of Clean Air Companies is an industry group that does not represent all specialty gas vendors uniformly.

> Proprietary Technology Protection – The original PGVP proposed that vendors using advanced, proprietary technologies would share their "best practices" with less technically competent competitors as a means of improving overall industry quality.

> PGVP Rule Notice and Comment – All gas vendors were not afforded sufficient opportunity to comment on the original rule.

Air Liquide took an opportunity to propose rule changes that would make the PGVP a better program, one that was more likely to achieve the desired goal of improving industry-wide quality. Some of the alternatives proposed included:

- Maintain an EPA-funded audit program to reduce end costs passed to customers.
- Make vendor participation in the PGVP optional in order to allow "market forces" to decide eventual success or failure of gas vendors.
- Require each facility producing EPA protocol gases to be quality certified via accreditation procedures such as ISO 17025 Certification (possibly EPA protocol-specific), A2LA Accreditation or some customized EPA Qualification Program.

EPA was initially reluctant to consider any option other than the original PGVP audit concept, so Air Liquide felt compelled to initiate legal action in 2008, seeking reconsideration of the PGVP's initial design and implementation. The court and EPA agreed that the rule's notice and comment requirements had not been sufficient, and so administrative reconsideration was granted. Throughout 2009-10, rule language suggestions were exchanged with EPA addressing alternatives to the original PGVP design, and in February of 2011, the modified version of the PGVP rule was promulgated.

2010 Blind Audit

How gas vendors typically measure up for EPA protocol gases

In 2010, EPA conducted their eighth protocol gas audit as a "dry run" test for the onset of the new PGVP. Using a format similar to prior audits, a total of 57 tri-blend protocol cylinders (171 components), containing the most commonly requested components, NO (NOx), SO_2 and CO_2 at low, medium and high ranges were purchased blindly from 14 gas suppliers. Verification analyses were performed by NIST. Samples were requested from three of Air Liquide's specialty gas facilities: Colorado, Texas and Michigan.

The full audit results showed similar variability in vendor performance as with prior audits: about 10.5% of the measured results exceeded the ±2% protocol accuracy requirement.

2010 EPA Blind Audit Results

			Low			Mid			High			
Producer	ST	Cyl	NO	SO_2	CO_2	NO	SO_2	CO_2	NO	SO_2	CO_2	Fail
Air Liquide	CO	3	0.65	0.60	0.14	0.35	0.47	0.26	0.31	0.36	0.10	0
	MI	3	2.57	0.44	0.63	0.09	0.15	0.21	0.24	0.00	0.33	1
	TX	3	0.21	0.84	0.01	0.81	0.09	0.08	0.36	0.08	0.26	0
Airgas	IL	6	1.34	1.90	0.82	0.34	0.08	2.66	0.37	0.16	0.54	1
	NC	3	0.23	0.22	0.37	1.16	0.08	0.47	2.22	0.20	0.06	1
	NJ	3	0.51	1.64	0.33	0.12	0.09	0.76	1.99	0.22	1.69	0
Linde	NJ	3	0.45	1.38	0.54	0.34	0.08	0.21	0.13	0.04	0.04	0
Liquid Technology	FL	3	2.26	2.03	3.24	2.53	2.50	0.97	2.55	2.90	0.49	6
Matheson Tri-Gas	OH	6	0.51	5.05	0.45	0.01	0.78	0.53	2.88	3.56	0.94	3
Praxair	CA	3	5.17	0.34	0.15	0.93	0.49	0.31	0.60	0.08	1.05	1
	PA	6	0.62	1.86	0.56	1.49	0.30	0.55	2.13	0.38	1.19	0
Red Ball Oxygen	LA	3	1.32	0.12	0.52	0.09	0.33	0.44	1.11	0.30	0.31	0
Scott-Marrin	CA	3	0.93	0.21	0.13	0.92	0.08	0.27	1.61	0.05	0.56	0
Specialty Air Technologies	CA	3	0.85	0.59	0.19	0.22	0.88	0.61	1.01	0.78	0.19	0
Specialty Gases of America	OH	6	0.85	0.69	0.24	0.75	0.45	0.38	1.09	0.17	0.92	0

Blue	Meets product claims: accuracy within ±1%
Orange	Meets protocol requirements (±2%), but fails ±1% accuracy claim
Red	Fails protocol requirements (±2%)
	Statistics require > 2.2% to fail audit
	MI low NO failure traced to O_2 contamination in blending

A truly rare occurrence – an Air Liquide component failure!

Cylinders of Air Liquide's SCOTT™ brand EPA protocol gases have historically flawlessly passed all of the previous seven EPA blind audits conducted up to 2010. In the 2010 audit however, one Air Liquide cylinder (out of nine) failed in the low NO concentration.

The failed cylinder was produced by our Troy, Michigan production facility and exceeded the allowed analysis difference of 2.2% by 0.37%. All other Air Liquide cylinders passed easily, including the other two cylinders produced by Troy.

We've since discovered that the low NO in the failed cylinder was not analyzed inaccurately, but rather, it had a slowly declining NO concentration. The decline rate was so slow that the cylinder passed analysis as being ±1% accurate, both at the time of production and after the required seven-day stability test. However, the NO value continued to decline after shipment, so that by the time the cylinder was analyzed by NIST, it had declined to the point where the NIST analyzed value and our original tag value differed by more than the allowable 2.2%.

Why our NO concentration declined

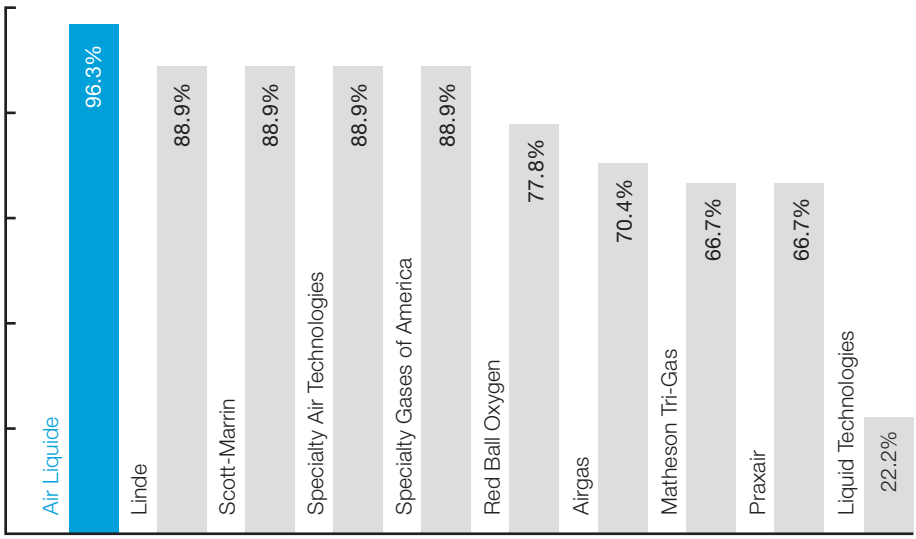
NO is a highly reactive molecule that, in the presence of O_2, converts to NO_2. The problem cylinder was contaminated with a trace of oxygen, affecting the NO but not the other components. Because of the low NO concentration, the problem cylinder had been blended on a station used for special, one-of-a-kind products. We have since traced the source of the O_2 to a cylinder evacuation manifold on this station, in which a slight leak allowed a trace amount of air to enter the cylinder during a pre-fill evacuation. More than 50 years of experience producing complex mixtures has taught us: this is all it takes to vitiate a precision gas mixture.

Good news for Air Liquide customers who depend on our accuracy and stability

Unlike the failures of other suppliers that indicate system-wide problems, Air Liquide's failure was an isolated anomaly, which did not involve our primary protocol-blending and analysis technologies. Besides repairing and recertifying the faulty manifold, we've retested and verified that each filling and evacuation manifold at every facility is leak-free. We've also added stringent measures to our preventative maintenance and performance evaluation procedures, making them the most comprehensive in the industry.

Air Liquide remains committed to quality and the highest possible accuracy

The technologies that deliver the world-class quality for which SCOTT™ brand products are well known are not only still in place, but also are more robust than ever. Our commitment to quality is evident in the data as shown in the table on the next page. Our Colorado and Texas facilities showed the smallest total variation (< 0.35%) from the NIST-analyzed values of any of the audited vendors. And if you discount the failed cylinder, our Michigan facility shows similar results.



Components Analyzed with 1% Accuracy

Air Liquide exhibits the highest overall accuracy by achieving the highest % of tests with 1% accuracy or better.

PGVP Going Forward

Air Liquide industry leadership achieves an improved and more equitable PGVP for all

Through the administrative reconsideration process, many of Air Liquide's concerns with the original PGVP rule were addressed. Some of the changes that resulted from Air Liquide's involvement are:

> PGVP Rule Notice and Comment – All vendors have now been given ample opportunity to comment.

> Technical Content – The small audit sample size (up to four cylinders per year) still persists; it is just not practical to collect a large enough "representative" sample of cylinders. However, EPA now disclaims and cautions customers not to draw conclusions about overall vendor quality from the results.

> Program Administration through ICAC – This provision has been eliminated.

> Unknown and Inequitable Program Fees – PGVP audit costs are now fixed and are much lower than originally estimated. The "per facility" PGVP fee assessment, which could have encouraged vendors to close facilities, is no longer an issue. In addition, the added costs of PGVP fees have been reduced to the point where the impact on the customer price is minimal.

> Proprietary Technology Dissemination – This provision has also been eliminated, thereby protecting the intellectual property of all vendors, spurring innovation and promoting increased competition among vendors via technology advancements.

While still not perfect, we at Air Liquide believe that, as a direct result of our efforts, the revised PGVP is greatly improved over what was originally proposed.

Protocol Gas Verification Program

Air Liquide now fully supports EPA's new PGVP, and all five of Air Liquide's specialty gas facilities are now fully registered EPA protocol gas production sites.

- Plumsteadville, Pennsylvania
- Troy, Michigan
- La Porte, Texas
- Longmont, Colorado
- Santa Fe Springs, California

At Air Liquide, we remain committed to producing the most accurate EPA protocol gases available in the industry. These include our SCOTT™ brand RATA Class™ EPA Protocol Gases with guaranteed ±1% analytical accuracy, and SCOTT brand Compliance Class™ Protocol Gases with ±2% analytical accuracy for standard regulatory reporting applications. Both mixture classes will continue to be blended using high performance technologies such as ACUBLEND™ and GRAVSTAT™, and analyzed using the latest Interference-Free™ instrumental analysis technology. SCOTT brand EPA protocol gases feature dual-analysis directly traceable to NIST reference standards.

PGVP participation

Registration of sites with EPA

- A contract with NIST for payment of services
- Demonstration of technical qualifications is not required

Production sites will be blindly audited once each year

NIST will analyze audit cylinders

- Analysis to assess accuracy to ±1%
- Audit findings reported within six months

Post-analysis disputes

- Production sites allowed 60 days to perform reanalysis or resolve other discrepancies on audit cylinders before NIST finalizes report

EPA publishes audit results on agency web sites

Any Part 75 source using EPA protocol gas must obtain the gas from a registered PGVP participant

Any Method 3A, 6C or 7E using EPA protocol gas for testing on a Part 75 source must obtain the gas from a registered PGVP participant

AIR LIQUIDE™

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Founded in 1902, Air Liquide is the world leader in industrial and medical specialty gases and related services, providing innovative solutions for the manufacture of everyday products and for the protection of life.