Gas Purity

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Does 99.9999%
mean there is only 1 ppm
of impurities?

Total Purity Paradox
(D. Krippene)

- Gas Purity is confusing when dealing with trade names or grades.
- Purities range from 98.0% to 99.99995% with no apparent standardization.
- Grade names for gases, unlike chemicals, are not regulated or characterized by any organization.
- Total Purity falls well short of characterizing the purity of specialty gases used for analytical instrumentation.

Purity Defined
(D. Krippene)

- **Total Purity by Assay:**
  - Purity obtained by comparison to "known" pure standard.
    - Used for medical oxygen to ensure minimum life sustaining
    - Acceptable "Go, No Go" test
    - Unacceptable for identification of critical contaminants
- **Total Purity by Difference:**
  - Purity obtained by subtracting "known" impurities from 100%.
    - Each impurity analyzed against individual standards
    - Currently accepted method for Semiconductor gases

Six Deadly Sins of Purity in Gases
(D. Krippene)

- Impurities are the same in both liquid and gas phase.
- Unit of Measure is not important.
- Sources of Contamination are treated equally by all suppliers.
- Number of Contaminants is universal for all suppliers.
- Lower Detectable Limits for Impurities analyzed are well established.
- Published Purities truly characterized the quality of a gas.

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Phase Analyzed
(D. Krippene)

- **Non-liquefied gases are essentially homogeneous.**
  - Volatilized impurities are considered equally distributed throughout gas matrix.
  - Does not address container issues of “bound” moisture, materials of construction bleed, or particles.
- **Liquefied Gases are non-homogeneous.**
  - Present unique sampling challenges.
  - High vapor pressure, low boiling impurities (atmospherics) concentrate in vapor phase.
  - Low vapor pressure, high boiling impurities (oils, water) tend to concentrate in the liquid.
  - Issues with containers particularly influential to liquid phase.
  - Impurities concentrate in liquid as gas is withdrawn.

Units of Measure

- Units Defined:

  Molar percent is based on constituent volume fractions. Five equal volume impurities of differing weights still constitute equal fractions.

  Weight percent is based on constituent weight fractions. Five differing weight impurities of similar volume will constitute different fractions.

- Most “gas phase” contaminants are typically measured and reported in volume of molar percent (ppm).
- Liquid contaminants are often, but not exclusively, measured and reported in weight percent (ppm).

Sources of Contamination

- **Three main sources of cylinder contamination.**
  1. **Manufacture: Impurities arising from production process.**
     - **CF_4 in Nitrogen Trifluoride.**
  2. **Cylinder Maintenance: Impurities arising from container.**
     - **Metallic particles from Carbon Steel, moisture from retest, residual from previous service.**
  3. **Cylinder Filling: Impurities arising from transfill process.**
     - **Lubricants, air, water, piping, valves, regulators.**

Number of Contaminants

- Single most important factor that influences purity.
- Specification of any material should reflect the expected impurities in a product.
- Total Purity by difference is adversely affected by the number of contaminants.
- Two products of similar quality, but different impurity specifications, often result in different purities.
- Fewer contaminants listed will give *perception* of higher quality (More for less).

Lower Detectable Limits (LDL)

- Limitation of analytical instrument and/or method by which concentration can be accurately quantified.
- Establishment of LDL extremely complex, and not universally shared between suppliers.
- Listing of LDL on Certificate of Analysis can adversely affect Total Purity by Difference.
  - Conservative method of subtracting the LDL value from 100%.
- LDL “hype” hides beneath a shroud of “proprietary”, closing scrutiny of methodology.

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Published Purities

- Specification *implies* a purity guarantee to end user.
- Most utilized qualification criteria, and most abused concept in the industry.
- Suppliers publish different specifications for similar products, causing confusion in industry.
- War of Words: *Typical* versus *Guaranteed*.
  - "Typical" by definition makes no guarantee of purity, and is simply an average concentration based upon actual data.

Playing the Game Units of Measure

- Tungsten Hexafluoride: Semiconductor Spec: 99.9%
  - Molar percent
  - Primary Impurity: HF @ 1000 ppm max.
- Tungsten Hexafluoride: Semiconductor Spec: 99.99%
  - Weight Percent
  - Primary Impurity: HF @ < 10 ppm.
- Cause of Disparity: *Weight* of HF molecule in perspective to *weight* of WF6 is magnitude order of difference.

Playing the Game Phase Analyzed

- Analysis of high vapor pressure, low boiling impurities in product specification labeled in “liquid phase”.
- Research Grade Carbon Dioxide.
  - O_2, N_2, CO impurities guaranteed for “liquid phase”.
  - Useless information since these concentrate as “non-condensables in vapor space.
  - Introduction of gas to tool will encounter “concentrated” impurity. Vapor to liquid ratios may be 10 to 1.
- Cause of Disparity: Inaccurate qualification by assuming homogeneity of impurities.

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Playing the Game
Sources of Contamination

- Specifications often *ignore* contaminants in a product, based on suppliers perception an impurity is irrelevant to end user's process.
  - Argon impurity in Nitrogen, Neon impurity in Helium
  - Hidden in sub-notes as *total inerts, excluding*…
- Specification is sometimes based on expected impurities in the "Bulk" source, ignoring affects of cylinder and transfill activities.
- Definition of *Batch* size inconsistent. Is it individual cylinder? One in Batch of 10, 20, 100? How often is batch analyzed? Each run? Once a year?

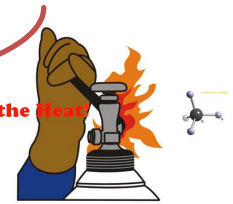
Playing the Game
Number of Contaminants

- Identified earlier as the single most important factor affecting total purity, it is the easiest manipulated to enhance purity.
- Total purity by difference is adversely affected by the number of contaminants listed.
- Deleting certain impurities may substantially improve total purity.

What You See Is What You Get…

Impurities (ppm)	Research	Carrier	Zero
Argon	15.0		
CO_2	0.5		
CO	1.0		
CH_4	0.5		
Krypton	10.0		
Nitrogen	15.0	15.0	
N_2O	0.1		
THC	**0.5**	**0.5**	**0.5**
Water	0.5	0.5	
Impurities By TPD	**43.1**	**16.0**	**0.5**
	99.996%	**99.998%**	**99.999%**

Is this really the most pure?



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Thank You

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