

**Intertek**

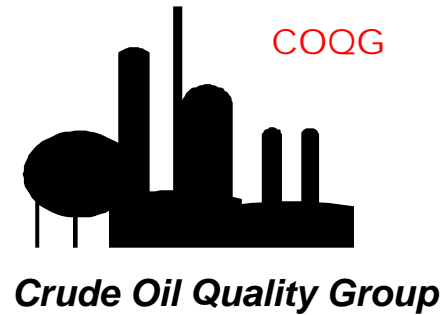
# Searching for Mercury

## COQG Meeting

New Orleans, LA

March 6, 2008

Tony Nguyen  
Laboratory Development  
Americas



## Presentation Overview

- Introduction to Intertek
- Mercury Measurement Techniques
- Recent Studies of **Sticky Mercury** in Hydrocarbon Production and Processing
- Impact of Extraction of Mercury from Sampling Containers
- Functional and Molecular Speciation

**Intertek**

# Worldwide Network

**We provide testing, inspection and certification services the world over**

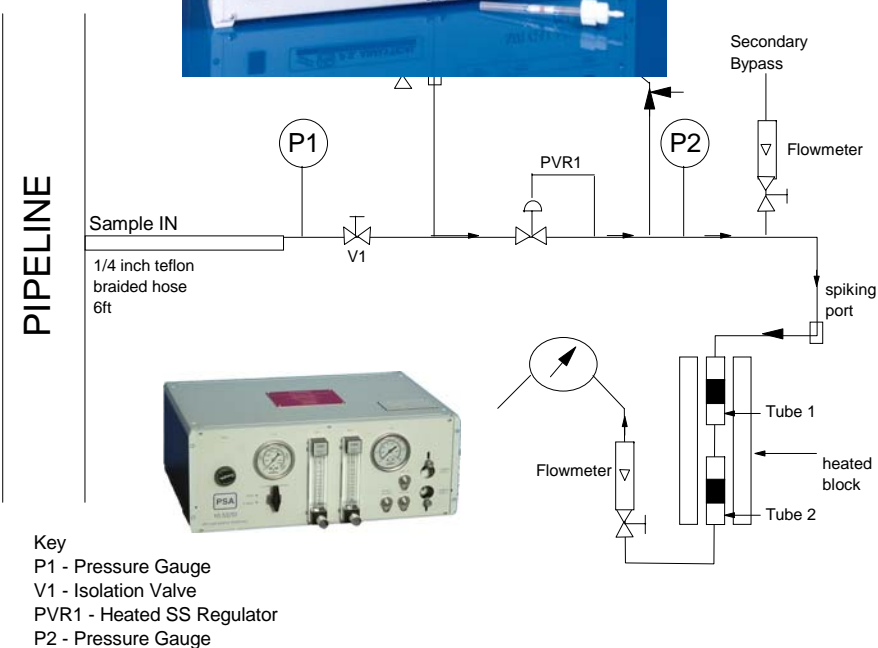


## Global Network

- 
- A light gray world map is positioned in the background, centered behind the text. It shows the outlines of continents and major countries.
- Laboratories 355
  - Offices 578
  - People 20,000
  - Countries 110
  
  - Floated May 2002
  - Market capitalization at 31st Dec 2006 £1.3bn
  - Corporate website [www.intertek.com](http://www.intertek.com)

## ISO 6978 Part II

- Scope
  - Natural Gas
- Equipment
  - Gold Amalgamation
  - Atomic Fluorescence (AFS) or Atomic Absorption (AAS)
  - Heated Manifold Sampling
- Detection
  - 0.001 ug/M3 – 1 ug/M3
- Precision
  - Not Given in Method



## JLPGA-S-07

- Scope
  - LPG
- Equipment
  - Gold Amalgamation
  - CVAAS
  - Heated Vaporization
- Detection
  - 0.01-1000 ng Hg mass
- Precision
  - Not Determined



## UOP 938

- Scope
  - Liquid Hydrocarbons
- Equipment
  - Nippon Instruments SP3D
- Detection
  - 0.1-10000 ng/ml
- Precision
  - Not estimated in the method
  - Calibrated by Serial dilution of traceable standards



Intertek

## LUMEX

- High Throughput
- Above 30ppb data equivalent to UOP 938
- Oil, Gas, and Water
- Zeeman Corrected CVAA



## **Speciation - Functional and Molecular**

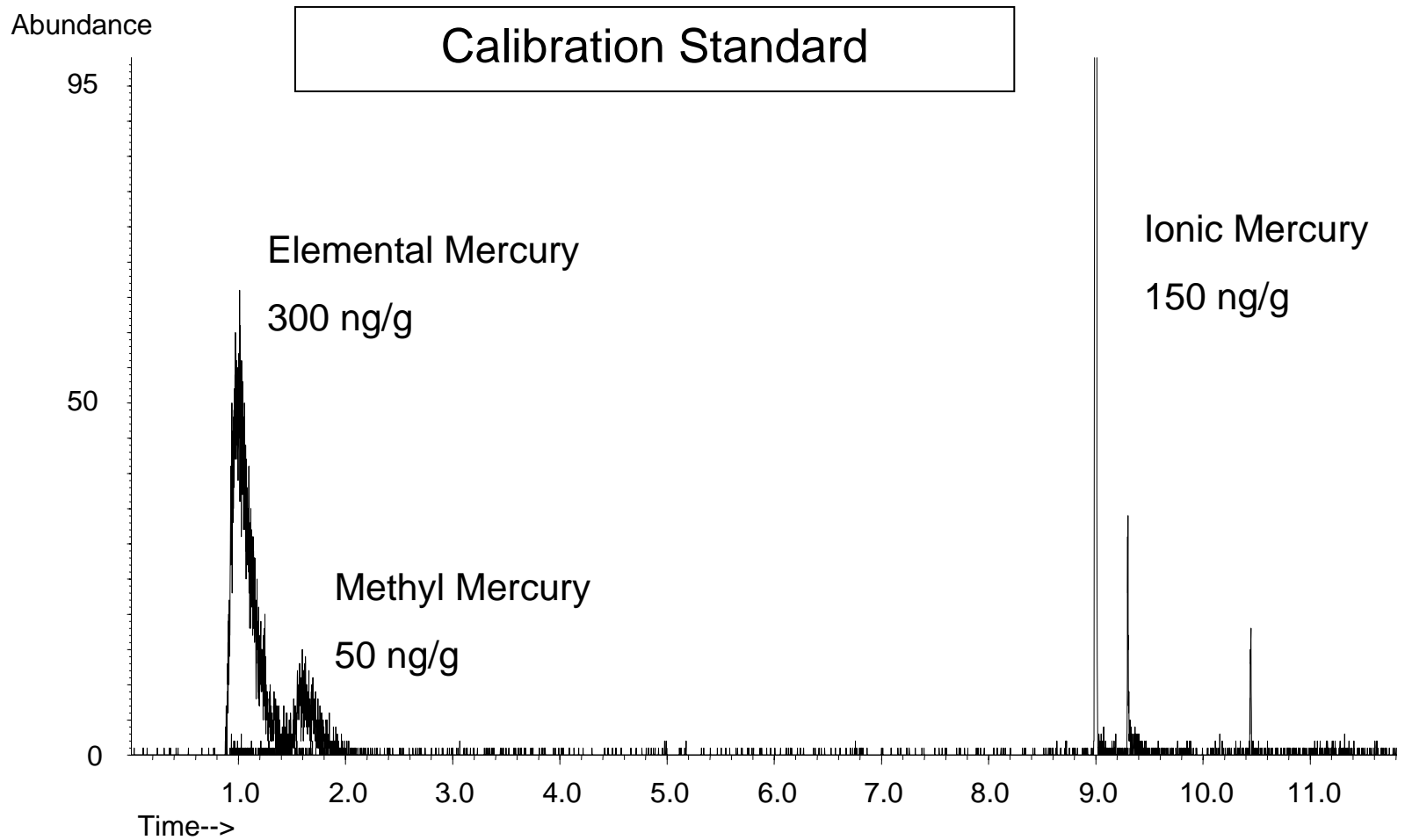
### Functional Speciation

- Volatile
- Particulates
- Water soluble
- Others

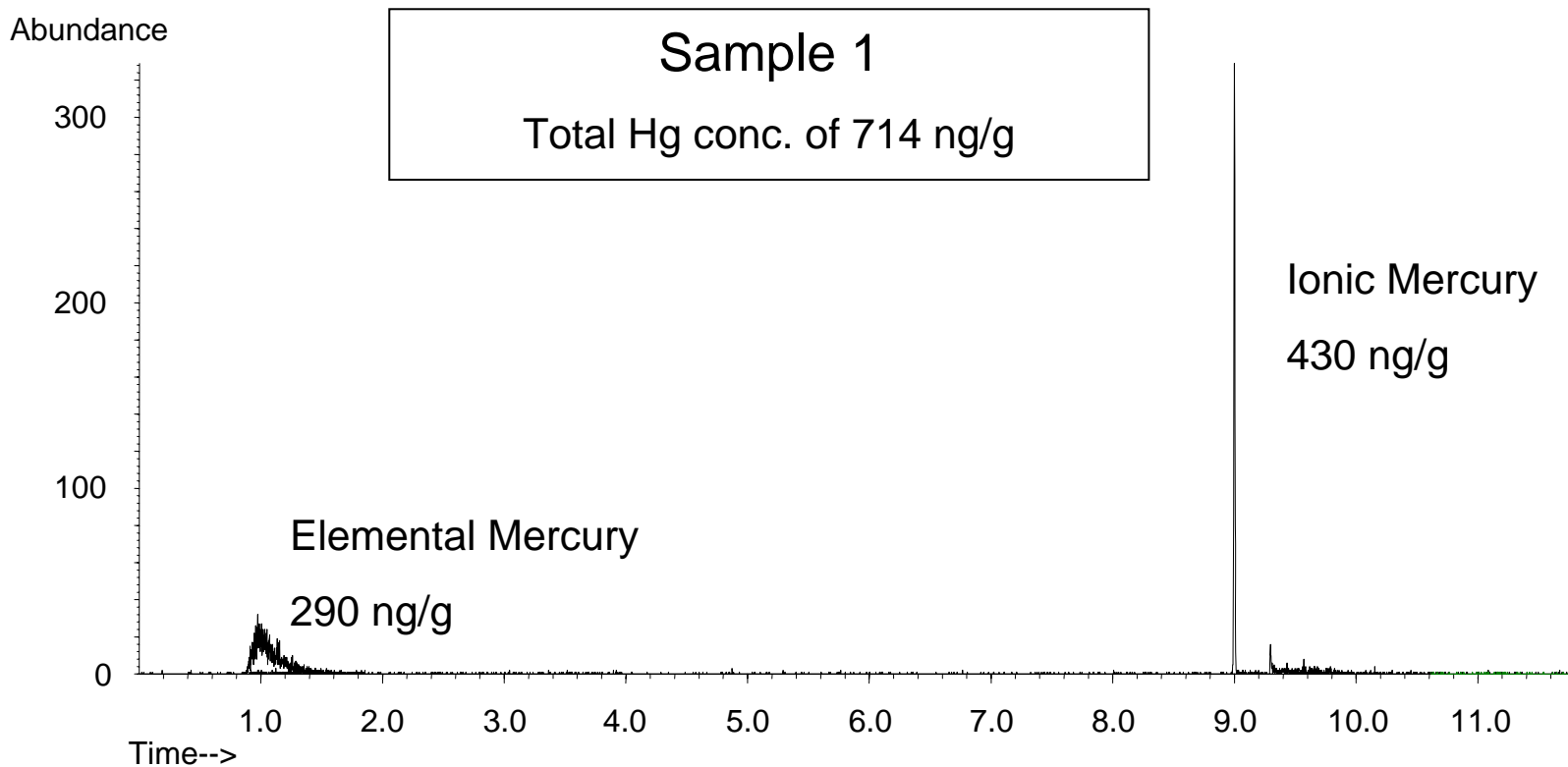
### Molecular Speciation

- GC-ICP-MS
- Elemental Mercury
- Methyl Mercury
- Ionic Mercury

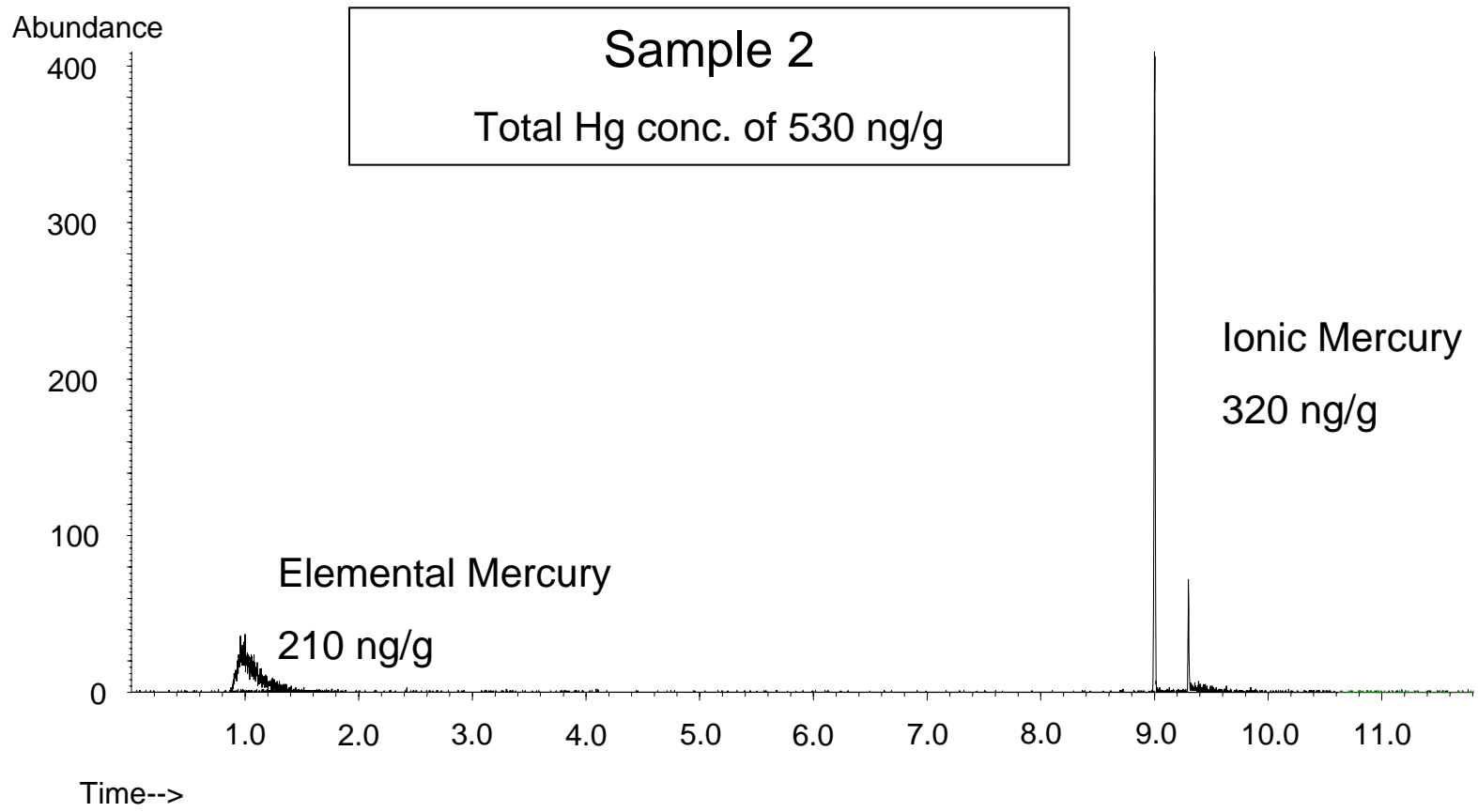
# Hg Speciation by GC-ICP-MS



# Hg Speciation by GC-ICP-MS



# Hg Speciation by GC-ICP-MS



## **Mercury Studies**

- **Mercury in Feedstocks, LPG, Naphtha, Condensate, and Effluent Water**
- **Transformation of Mercury Types**
- **Functional Speciation**
- **Refinery And Plant Mass Balances**
- **Mercury Removal Process Planning/Troubleshooting**
- **Liquid Asset Mercury Distribution Studies**
- **Mercury Adsorption Studies**

## Mercury Mass Balance Refinery Study

- Issue: Refining: On Site Mercury Study  
*Poor Mass Balance*
- Investigation: Sampling Containers -  
Potential adsorption of Hg onto glass surfaces.
- Result: Better Mass Balance  
Most samples must be measured  
both in liquid and on container surface

## Extraction of Adsorbed Hg Petroleum Distillate in Glass VOA Vials

	Hg in Liquid ppb wt	Extracted Hg ppb wt	Total Hg ppb wt
1	22	895	917
2	24	894	918
3	8	871	879
4	5	811	816

## Mercury Mass Balance Laboratory Distillation

- Issue: Hg Distribution Study  
Poor Mass Balance on  
Distillate Cuts From ASTM D2892
- Investigation: Mercury Adsorption on Glass  
Distillation Column
- Result: Significant Hg recovered  
from distillation column.  
Improved mass balance

## Distribution Study Closure Investigation

Sample	Total Hg (ng)	% Hg Recovered
Whole Crude	2,429,700	
Distillate Cuts	484,842	22.94
Acid Wash 1	250,000	
Acid Wash 2	125,000	
Acid Wash 3	75,000	38.50
Amino Acid 1	524,500	
Amino Acid 2	6,500	
Amino Acid 3	4,500	
Strong Oxidizer	28,000	61.67

## **Crude Oil – Hg recovered from glass Sample Containers**

- **Issue:** Total Hg measurements in known high mercury crude oil
- **Investigation:** Mercury Adsorption on glass containers.
- **Result:** Extraction of glass recovered additional 300 ng/g (30%) Hg

## Total Hg in Crude Oil Sample after Extraction

Hg in liquid hydrocarbon	1037 ppb wt.	
Extracted Hg using strong oxidizer	312 ppb wt.	30.1% increase
Total Hg in Crude	1349 ppb wt.	

## **Hg Measurements and Extractions Glass and Teflon Sampling Containers**

- **Issue:** How much Hg adsorbs to Teflon v. Glass
- **Investigation:** Investigate analytical techniques to measure extracted and total Hg on representative refinery distillate.
- **Result:** Consistent Total Hg results obtained with either extraction from Teflon or Glass. More repeatable extraction obtained from Teflon.

## Total and Extracted Hg VOA & Teflon Containers Petroleum Distillate

	VOA Bottles			Teflon Bottles		
Liquid Phase	14.10	16.62	15.92	16.04	15.68	16.12
Extraction	6.07	2.79	4.74	3.26	3.76	3.86
Total Hg (ppb wt)	20.17	19.41	20.67	19.30	19.45	19.99

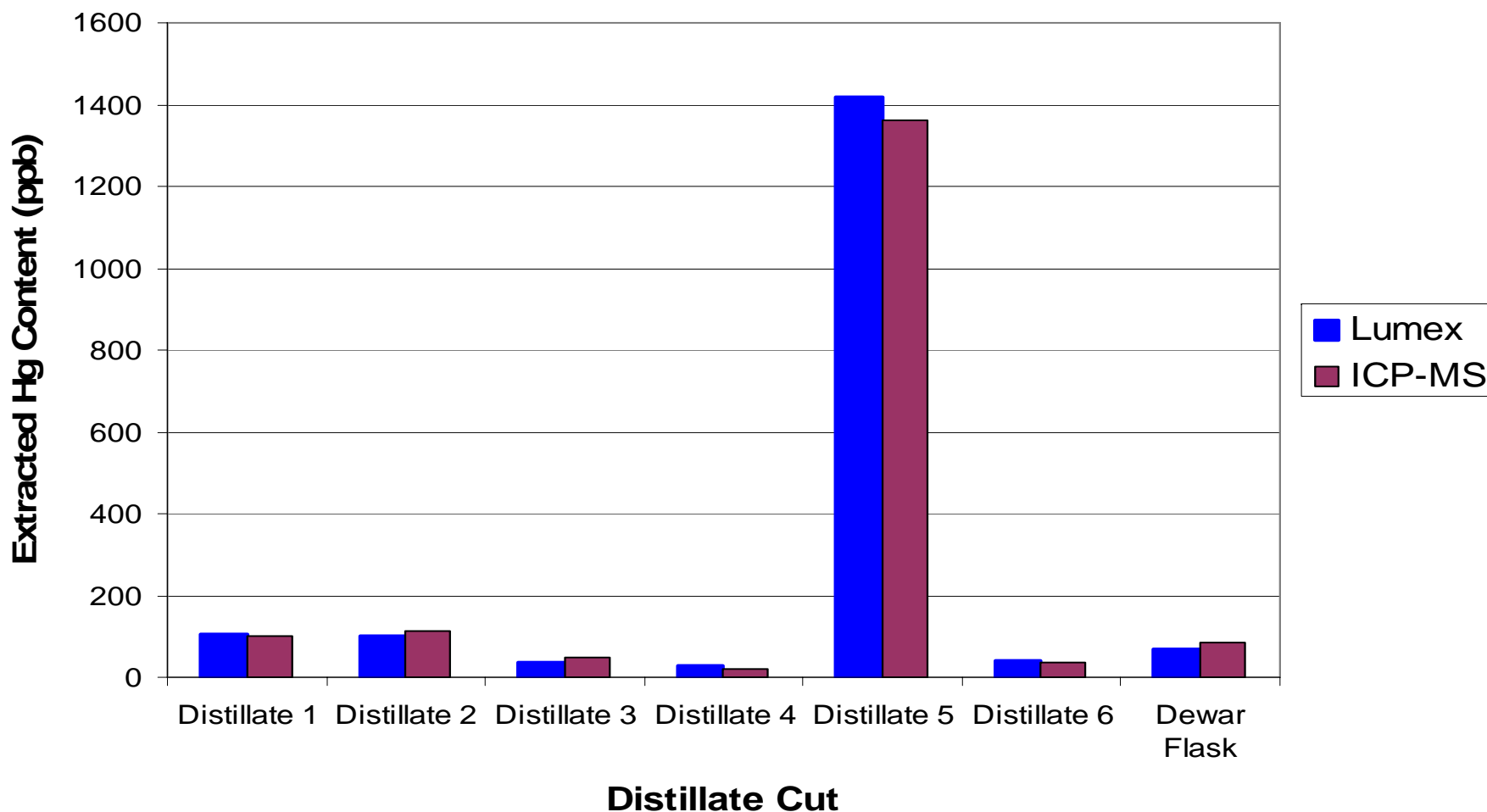
## **Hg Measurements on Hydrocarbon Assets As Received at the Refinery**

- **Issue:** Inconsistent Hg results from samples as received
- **Investigation:** Investigate sampling techniques to ensure representative sampling event at the refinery
- **Result:** Consistent results obtained with improved attention to sampling and extraction of Hg in the sample containers

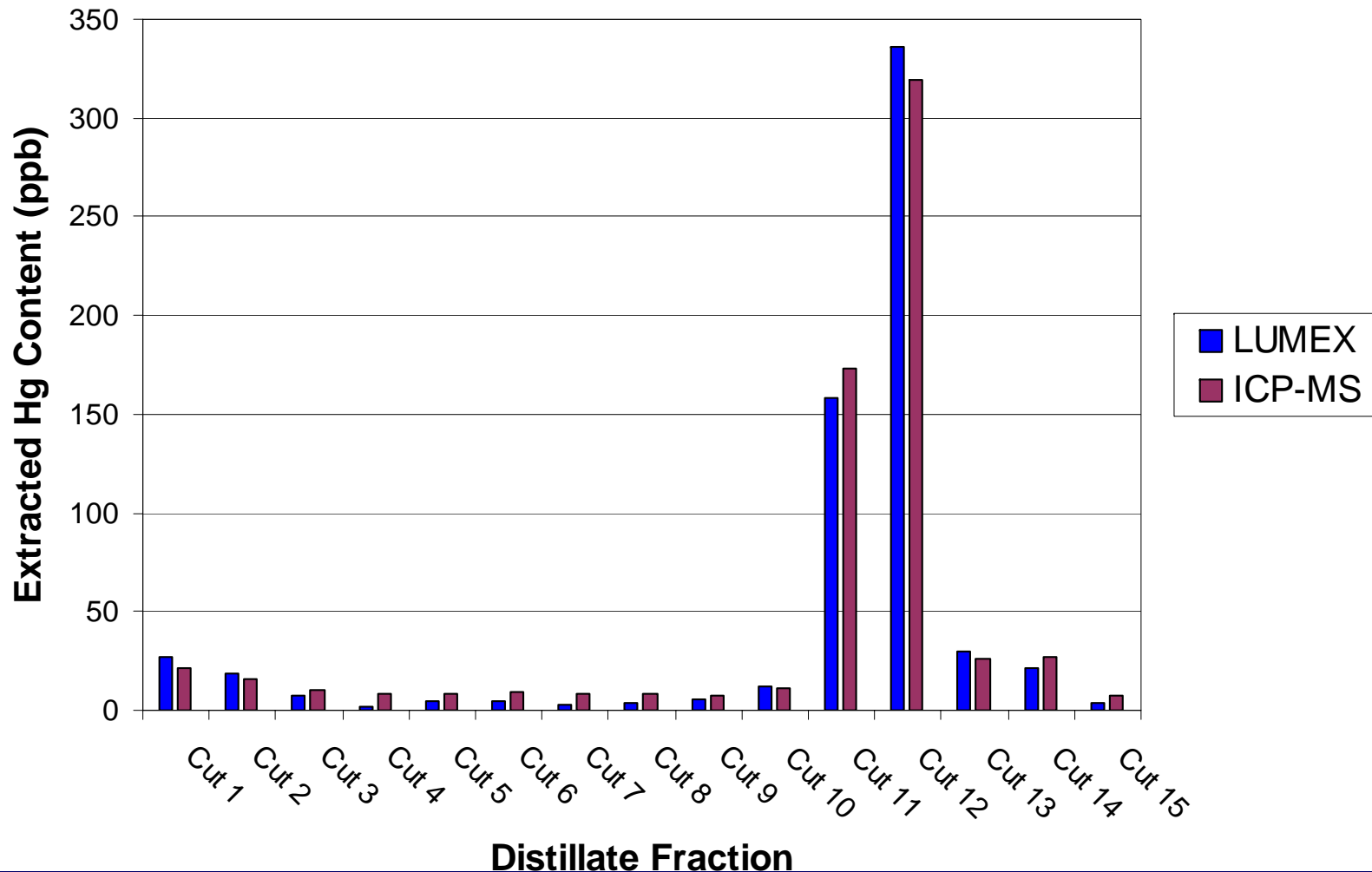
## Effects of Extraction on Hg Measurements in Liquid Condensate

Sample	Sample Container	Hg Result ppb wt	Total Hg ppb wt
1	1 L Glass Schott Duran	135	163
2	1 L Glass Schott Duran	129	166
3	1 L Unlined Metal Can	95	-
4	1 L Unlined Metal Can	67	-

# Corroborative Analyses Confirm “Sticky” Mercury Adsorbed to Sample Receivers



# Corroborative Analyses Confirm “Sticky” Mercury Adsorbed to Sample Receivers



## Summary

- The existing databases for liquid assets in our industry do not include “sticky” mercury and should be updated.
- Intertek has developed techniques to allow accurate measurements of total mercury in hydrocarbon liquids including the mercury sticking to the sample container.
- Corroborative analytical techniques provide insight into analytical uncertainty.
- Re-evaluate all mercury data if it does not include this newly confirmed phenomenon of “sticky” mercury.
- Intertek is the leading provider of vital analytical technology and experience globally.

## **Contact Us**

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