



Domtar Inc. Research Centre  
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## **CERTIFIED REFERENCE MATERIALS OF COAL-TAR PITCH**

### **PITCH-A, PITCH-B, PITCH-C AND PITCH-D**

These certified reference materials (standards) of coal-tar pitch were prepared by the Analytical Sciences Group of the Domtar Inc. Research Centre in Senneville (Montréal), Québec. The material is intended primarily for the determination of sulphur and trace elements in pitches, heavy bottoms and asphalt-type samples by X-Ray Fluorescence Spectrometry and various other techniques, such as atomic absorption (AA), plasma spectroscopy (ICP), etc. The certified samples were chosen with respect to sulphur concentration (high-low) and different levels of trace impurities in order to provide suitable calibration ranges for most common impurities.

#### **ORIGIN AND PREPARATION OF THE SAMPLES**

The bulk of the samples used for the certification was taken from a large (10 ton) plant sample. These pitches were thoroughly mixed, quartered and mixed again. Their homogeneity was tested by X-Ray Fluorescence Spectrometry. About 40 samples were taken from the bulk at random for the round-robin analyses, which were carried out in triplicate.

#### **ANALYTICAL METHODS USED**

The values given for sulphur and most of the trace elements were determined by round-robin directly on the air-dried (25EC) sample. The round-robin analyses were carried out by a variety of modern analytical methods. These involved INAA (Instrumental Neutron Activation Analysis), IPAA (Instrumental Photon Activation Analysis), X-Ray Fluorescence Spectrometry on ash and directly on the pitch, PIXE (Particle Induced X-Ray Emission) directly on the pitch, AA (Atomic Absorption) spectrophotometry on ash, photometry, LECO combustion apparatus with infra-red sulphur detection system, classical gravimetry, Mettler softening point determination and several others.

#### **STABILITY OF THE SAMPLE AND INSTRUCTIONS FOR DRYING**

The long term stability of the samples has not been rigorously tested. It is known from experience that the pitches should remain stable for several years. The results of the thermal analysis scans (DTA-TGA) carried out in an air atmosphere indicated the first sign of decomposition (weight loss) at about 140EC. It is recommended that the sample be kept in a tightly closed bottle and away from the sunlight and sources of heat.

#### **PACKAGING**

The samples are packaged in 60 g quantities. Glass bottles were used to prevent any contamination.

#### **DRYING**

The sample is supplied air-dried at 25EC. No additional drying is necessary, unless condensation occurs in the bottle. In this case, the sample should be dried in a desiccator using silica-gel or other mild drying agents.

#### **INORGANIC CONSTITUENTS IN THE COAL-TAR PITCH**

The mean concentrations given below are in micrograms/gram (ppm) unless stated otherwise. These refer to the pitch as bottled (no drying is necessary). The values in parenthesis correspond to standard deviations.

# **CERTIFIED ELEMENTS**

<b>ELEMENT</b>	<b>PITCH-A</b>	<b>PITCH-B</b>	<b>PITCH-C</b>	<b>PITCH-D</b>
<b>% S</b>	0.49 (0.03)	0.52 (0.03)	4.46 (0.09)	0.58 (0.03)
Al	245 (7)	228 (10)	9 (2)	1.2 (0.2)
Ca	91 (9)	41 (6)	3 (0.1)	1.4 (0.2)
Cl	118 (18)	122 (20)	18 (4)	1.3 (0.3)
Cr	0.87 (0.07)	1.1 (0.1)	0.4 (0.1)	2.2 (0.2)
Fe	200 (2)	280 (10)	14 (2)	4 (0.3)
Mn	2.7 (0.2)	3.3 (0.2)	0.21 (0.01)	0.030
Na	257 (44)	150 (25)	10 (3)	9 (2)
Ni	2.5 (0.1)	-	76 (6)	-
P	10 (3)	3 (0.5)	236 (20)	1 (0.2)
Pb	91 (4)	80 (3)	1 (0.1)	0.6 (0.1)
Si	358 (30)	408 (20)	20 (5)	10 (4)
Ti	18 (4)	16 (4)	19 (4)	0.32 (0.02)
V	1.2 (0.03)	0.89 (0.02)	170 (10)	0.06 (0.01)
Zn	88 (11)	90 (12)	1 (0.2)	1 (0.2)

# **OTHER VALUES AND ELEMENTS**

**(As Reported by the Participating Laboratories)**

SOFT POINT (EC)	115 (1)	118 (0.7)	129 (1.5)	86.5 (0.7)
ASH (950EC)	0.27 (0.01)	0.22 (0.01)	0.19 (0.02)	0.04 (0.01)
%C	94.0	93.4	83.4	92.7
%H	4.0	4.3	10.31	4.8
As	-	9	0.18	2.2
Br	1.7	4.8	0.25	0.08
Cd	-	2.5	<0.05	<0.5
I	0.33	0.6	1.4	0.84
K	43	34	2.2	0.6
Mα	17	<30	<16	<2
Sb	-	0.57	0.03	0.014
Sn	-	3.7	<0.7	<0.2

" - " Value not available.

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