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TRACE ANALYSIS IN QUARTZ(U) EAGLE-PICHER INDUSTRIES INC
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F19628-81-C-0072

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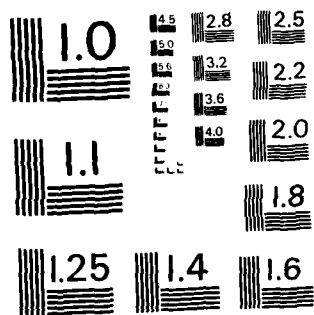
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RADC-TR-84-51
Final Technical Report
March 1984



TRACE ANALYSIS IN QUARTZ

Eagle-Picher Industries, Inc.

A. L. Fluesmeier

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Air Force Systems Command
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RADC-TR-84-51 has been reviewed and is approved for publication.

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SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED		1b. RESTRICTIVE MARKINGS N/A	
2a. SECURITY CLASSIFICATION AUTHORITY N/A		3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; distribution unlimited	
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE N/A			
4. PERFORMING ORGANIZATION REPORT NUMBER(S) N/A		5. MONITORING ORGANIZATION REPORT NUMBER(S) RADC-TR-84-51	
6a. NAME OF PERFORMING ORGANIZATION Eagle-Picher Industries, Inc. Specialty Metals Division		6b. OFFICE SYMBOL (If applicable) ESME	
7a. NAME OF MONITORING ORGANIZATION Rome Air Development Center (ESME)			
6c. ADDRESS (City, State and ZIP Code) P.O. Box 1090 Miami OK 74354		7b. ADDRESS (City, State and ZIP Code) Hanscom AFB MA 01731	
8a. NAME OF FUNDING/SPONSORING ORGANIZATION Rome Air Development Center		8b. OFFICE SYMBOL (If applicable) ESME	
9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER F19628-81-C-0072			
6c. ADDRESS (City, State and ZIP Code) Hanscom AFB MA 01731		10. SOURCE OF FUNDING NOS.	
		PROGRAM ELEMENT NO. 62702F	
		PROJECT NO. 4600	
		TASK NO. 17	
		WORK UNIT NO. 49	
11. TITLE (Include Security Classification) TRACE ANALYSIS IN QUARTZ			
12. PERSONAL AUTHOR(S) A. L. Fluesmeier			
13a. TYPE OF REPORT Final		13b. TIME COVERED FROM Jul 81 to Dec 83	
14. DATE OF REPORT (Yr., Mo., Day) March 1984		15. PAGE COUNT 24	
16. SUPPLEMENTARY NOTATION N/A			
17. COSATI CODES		18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)	
FIELD 07	GROUP 02	SUB. GR. Quartz Crystals Chemical Analysis Atomic Absorption	
19. ABSTRACT (Continue on reverse if necessary and identify by block number) The objective of this work was to devise a technique capable of determining trace impurities in quartz at the parts per million range. The technique selected was atomic absorption and a procedure was devised for determining lithium, sodium, potassium, iron, aluminum and carbon. Over 150 quartz samples were analyzed using this technique.			
20. DISTRIBUTION/AVAILABILITY OF ABSTRACT UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS ☐		21. ABSTRACT SECURITY CLASSIFICATION UNCLASSIFIED	
22a. NAME OF RESPONSIBLE INDIVIDUAL Alton F. Armington		22b. TELEPHONE NUMBER (Include Area Code) (617) 861-4834	
		22c. OFFICE SYMBOL RADC (ESME)	

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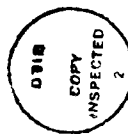
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This final report covers the work performed on Department of the Air Force Contract No. F19628-81-C-0072 by the Analytical Department of Eagle-Picher Industries Inc., Miami, Oklahoma and covers a period of time from July 1, 1981 through December 31, 1983.

I LITERATURE SURVEY

The first part of this contract was the performance of a literature survey of analytical methods for trace analysis in quartz and fluoride glasses.

During this search the Dialog on-line retrieval service was used. This service allows many sources to be checked in a relatively short time.

The literature for the period 1972-1981 inclusive dealing with quartz and fluoride glasses was surveyed using the Chemical Abstracts data base. Key words which were used in the broadly defined search were the following: quartz, impurity, silica, lithium, sodium, potassium, aluminum, fluoride, fluoride glass, analysis, determination and amorphous. A total of more than 2100 articles were identified about 10% of which were pertinent to the project. It is not surprising that most of the analytical references found were for impurity levels much higher than we were interested in. It therefore became necessary to develop an analytical procedure suitable to our purposes. The results of the literature search were forwarded to the

contract monitor. The analytical procedure developed in this laboratory was used for subsequent analytical work on this contract. A copy of this procedure can be found in Appendix I.

II ANALYSIS OF SAMPLES

A total of 205 samples were analyzed on this contract. These included not only samples of quartz and quartz-growth related materials but also samples of Lithium Niobate, Indium, Phosphorous and Indium Phosphide. Almost all of the samples were analyzed using the analytical procedure developed in this laboratory. (See Appendix I) Some of the special samples, however, were analyzed by Emission Spectrography using a standard laboratory procedure.

The analytical procedure developed in this laboratory proved to be very good in that it allowed very little opportunity for sample contamination. The limiting factor in this procedure is not the dissolution of the sample but the final detection of the required elements. Atomic absorption was used during this contract. This limited our detection limits due to sample dilution. We intend to use an Inductively Coupled Plasma/Mass Spectrometer on these and similar samples in the future. We expect this new state-of-the-art instrument to lower our detection limits for these elements to the parts-per-billion range.

Analytical results on all of these samples are reported in Tables I through Tables VII.

Table I
AF Contract F-19628-81-C-0072
Samples for Analysis

No.	'81 Date	Identi.	μg/g									
			Al	Fe	Na	Li	K	C	Ag	Pt	Ge	
1.	7/20	QP-5	9.	4.	8.	6.	0.7	<40.				
2.	7/27	X-23	453.	10.	11.	126.	8.				<1.	
3.	8/24	QA8-B	1.3	1.7	6.3	0.8	<0.5					
4.	8/24	QA8-T	0.7	1.7	5.2	<0.5	<0.5					
5.	8/24	X-33	6.9	2.7	9.4	1.7	0.7					
6.	9/29	F-2	7.	44.	50.	6.	1.	<40.				
7.	9/29	QA-6	17.	4.	5.	5.	<0.5					
8.	9/29	MOT	16.	<0.5	2.	5.	<0.5					
9.	9/29	SAR	28.	0.5	14.	3.	<0.5					
10.	9/29	Solution	44.									
11.	10/21	F-2 ReRun	6.	9.	16.	2.	<0.5					
12.	10/21	PQ-G-2	4.	0.8	108.	1.	<0.5					
13.	10/21	QA9-T	2.	<0.5	4.	<0.5	<0.5					
14.	10/21	QA9-B	2.	0.8	1.	0.6	<0.5					
15.	10/21	QA10-T	3.	0.9	0.5	<0.5	<0.5					
16.	10/21	QA10-B	2.	1.	4.	<0.5	<0.5					
17.	10/21	X-34	47.	3.	34.	12.	<0.5					
18.		QA-3	18.	561.	42.				18.			
19.		X-12	96.	444.	122.				2.7			
20.		QP-1	22.	2870.	28.	8.	2.7		<10.			
21.		QP-2	8.	708.	<1.	93.	8.1		<10.			
22.		QA-5	13.	30.	<1.	<1.	<1.					
23.		QA4-P	15.	2590.	23.	1.8	7.2					
24.		Sand	7.	330.	<1.	1.	4.					
25.		F-2	10.	690.	10.	2.8	2.6					
26.		ANISR	<2.	420.	10.	<1.	3.8					
27.		INC	15.	0.26	3.1	0.75	1.5					
28.		NC	32.	2240.	17.	4.3	4.					
29.		NS	55.	1500.	27.	9.7	4.					
30.		X-23	388.	6.4	3.6	100.	<1.					
31.		QA-7	5.4	2.2	1.	1.3	<1.					
32.		X-32	68.									

Table I Continued

No.	'82 Date	Identification	$\mu\text{g/g}$										Pt	Ge
			Al	Fe	Na	Li	K	C	Ag					
33.	2/9	1	9.8	2.2	20.	0.8	3.1							
34.	2/9	2	7.9	1.7	32.	0.5	5.7							
35.	2/9	3	27.	4.1	69.	1.4	10.							
36.	2/9	4	33.	3.0	24.	2.7	6.5							
37.	2/26	12-B	6.	2.0	2.4	2.4	1.							
38.	2/26	14-T	5.	27.0	0.6	11.	0.5							
39.	2/2	Metal Screw	400. major	31.			17.							
40.	2/2	Metal Piece	390. major	740.			110.							
41.	5/11	Never Seize	1-5%											
42.	5/26	QA16B	9.	0.9	28.	3.	<0.5							
43.	6/1	QA18	<0.5	<0.5	0.5	<0.5	<0.5							
44.	8/30	Mot. 1	21.	2.7	10.	2.5	3.4							
45.	8/30	Mot. 2	6.	<0.5	8.3	1.3	<0.5							
46.	8/30	Mot. 3	3.9	2.5	890.	2.8	<0.5							
47.	8/30	Mot. 4	<0.5	<0.5			1.6							
48.	8/30	Mot. 5	2.1	5.8			4.6							
49.	8/30	Mot. 6	<0.5	<0.5			0.7							
50.	8/30	Mot. 7	2.3	3.1			3.6							
51.	8/30	Mot. 8	<0.5	<0.5			3.7							
52.		Amethyst	35.	91.	6.4	15.	124.							
53.		Citrine	13.	148.	22.	15.	857.							
54.		14.31A0Z	1.3	<0.5	21.	1.4	<0.5							
55.		SARP 1	1.2	3.0	15.	0.9	<0.5							
56.		SARP 2	5.7	2.1	6.7	1.9	<0.5							
57.		SARP 3	9.9	4.0	4.1	3.0	<0.5							
58.		QA22X	51.	1.6	12.	24.	<0.5							
59.		QA22Z	35.	1.2	11.	17.	<0.5							
60.		14.56-1	4.4	0.9	21.	2.5	<0.5							
61.	2/24/83	Mot. 1	3.8	1.2	5.6	0.8	<0.5							
62.	2/24/83	Mot. 2	5.3	1.8	1.6	2.5	0.9							
63.	2/24/83	Mot. 3	3.3	0.5	1.7	0.8	<0.5							

Rerun of new sample

Table I Continued

No.	Date	Identi.	μg/g										Ge
			Al	Fe	Na	Li	K	C	Ag	Pt			
64.	2/24/83	Mot. 4	2.1	1.1	1.1	0.6	< 0.5						
65.	2/24/83	Mot. 5	0.6	<0.5	<0.5	<0.5	< 0.5						
66.	2/24/83	Mot. 6	4.2	0.6	7.1	1.4	< 0.5						
67.	2/24/83	Mot. 7	23.	9.	11.	6.9	< 0.5						
68.	2/24/83	Mot. 8	1.7	3.3	2.5	0.7	< 0.5						
69.		GG-2-48	1.0	2.6	1.4	<0.5	< 0.5						
70.		Z-Growth	7.5	1.0	2.2	2.0	< 0.5						
71.		QA21X	5.3	<0.5	0.9	1.5	0.5						
72.		QA21Z	15.	<0.5	1.2	4.0	< 0.5						
73.		QA20X	28.	0.5	3.3	7.6	< 0.5						
74.		QA20Z	17.	0.8	1.1	4.7	< 0.5						
75.		X39B	6.5	3.2	2.1	1.5	< 0.5						
76.		X39T	3.7	1.4	1.2	0.9	< 0.5						
77.		QA24Z	3.8	<0.5	<0.5	1.0	< 0.5						
78.		QA25X	13.	<0.5	2.7	4.4	< 0.5						
79.		X39X	6.4	0.9	0.9	1.3	< 0.5						
80.	4/12/83	QA26BZ	43.	.8	14.	11.	1.4						
81.	"	X39Z	3.6	2.7	1.5	1.	< .5						
82.	"	X43BZ	33.	1.8	5.4	8.7	2.8						
83.	"	X44	4.2	.9	3.7	1.2	< .5						
84.	"	Z Mined Mat.	28.	1.7	2.0	7.5	.8						
85.	"	QP-10	9.7	5.6	2.2	2.6	1.1						
86.	"	Material from											
		Wall	920.	22.%	6.6%	127.	32.						
87.	5/16/83	Z Seed FMQA-29	1.2	1.6	4.4	0.5	1.9						
88.	"	FM Z Seed X37	3.3	1.4	7.8	0.9	< 0.5						
89.	"	Mot Z Mined											
		Quartz	3.1	3.7	7.7	1.2	9.6						
90.	5/31/83	Z Mined											
		Nutrient	7.0	<0.5	8.6	2.3	5.2						
91.	6/1/83	No ID	2.3	<0.5	6.1	0.6	6.8						
92.	"	Nutrient Z	1.6	<0.5	8.5	1.1	8.8						

Table I Continued

No.	Date	Identi.	$\mu\text{g/g}$									
			Al	Fe	Na	Li	K	C	Ag	Pt	Ge	
93.	7/11/83	QP-15	3.9	2.1	9.8	0.7	6.3					
94.	"	X-35	3.6	2.5	18.	1.5	<0.5					
95.	8/22/83	Dynasil Glass										
	"	Soaked	4.5	2.5	2.8	<0.5	3.7					
96.	"	Dynasil Glass										
	"	Untreated Clear	3.3	2.3	2.1	<0.5	1.2					
97.	"	Dynasil Glass										
	"	Untreated yellow	1.0	1.0	1.7	<0.5	1.7					
98.	"	Dynasil Glass										
	"	Cleaned	1.0	<0.5	<0.5	<0.5	<0.5					
99.	"	X-47 Nutrient	9.1	<0.5	1.3	3.6	1.3					
100.	"	Quartz no ident.	4.0	<0.5	1.3	1.2	<0.5					
101.	9/6/83	GC 6/21	8.3	1.3	4.7	2.1	1.7					
102.	9/9/83	Soln. from										
	"	52ET11	2.2	0.4	-	-	-					
103.	"	Sol. from QA-30	1.3	0.5	-	-	-					
104.	"	QP-17 Potassium	3.0	3.4	4.4	1.0	1.3					
105.	9/20/83	GC11; T2-10 Mot.	2.8	0.7	1.9	1.0	<0.5					
106.	"	GC11; T10-10	"	2.7	3.2	0.9	<0.5					
107.	"	GC12; T1-2	"	<0.5	8.5	1.0	1.2					
108.	"	GC12; T5-1	"	1.8	8.0	1.1	<0.5					
109.	"	GC13; T1-7	"	2.6	2.8	0.9	2.5					
110.	"	GC13; T3-1	"	1.7	0.8	0.8	<0.5					
111.	"	GC14; T5	"	0.9	0.6	2.2	<0.5					
112.	"	GC15; T5	"	0.5	<0.5	1.5	<0.5					
113.	"	GC16; T1-9	"	3.4	0.6	6.4	1.0					
114.	"	GC16; T2-9	"	3.6	<0.5	2.1	0.9					
115.	"	GC17; T2-7	"	3.3	<0.5	1.2	0.8					
116.	"	GC17; T2-5	"	4.1	<0.5	1.3	1.3					
117.	"	GC18; T5	"	<0.5	<0.5	0.7	<0.5					
118.	9/23/83	Quartz 1	"	3.2	1.1	396.	0.8					
119.	"	Quartz 2	"	2.7	1.0	342.	0.8					
120.	"	Quartz 3	"	1.1	1.1	287.	<0.5					

Table I Continued

No.	Date	Identi.	$\mu\text{g/g}$									
			Al	Fe	Na	Li	K	C	Ag	Pt	Ge	
121.	"	Soln. 4	" <0.5	-	-	-	<0.5					
122.	11/3/83	Z Growth	3.4	<0.5	1.7	0.7	<0.5				1100.	
123.	"	X-46A	0.8	0.5	2.3	<0.5	<0.5				< 10.	
124.	"	Comm. Seed										
125.	"	Etched	1.2	1.7	1.4	<0.5	4.1					
		SY2ET7 Seed										
126.	11/2/83	Non Etch.	5.1	2.1	9.9	1.8	6.0					
		X49 X mined	14.0	0.5	1.6	3.4	0.8				2200.	
127.	"	X49 Z mined	1.9	<0.5	0.7	<0.5	<0.5				2170.	
128.	11/28/83	X53 low	30.0	0.9	6.8	8.6	0.9				3310.	
129.	"	X53 surface	26.2	2.1	6.4	8.0	<0.5				3820.	
130.	12/6/83	QP-16 Z mined	3.4	0.5	5.6	<0.5	5.0					
131.	"	QP-18 Z mined	1.1	<0.5	1.4	<0.5	<0.5					
132.	12/12/83	QA31 X	<0.5	0.9	<0.5	<0.5	<0.5					
133.	12/12/83	X48	1.4	1.8	1.0	<0.5	0.5					
134.	"	X50A	<0.5	<0.5	1.9	<0.5	2.2					
135.	"	X50B	0.9	0.8	1.5	<0.5	<0.5					
136.	"	X51X	10.3	0.5	45.	3.6	0.8					
137.	12/19/83	14.180-4										
		Sawyer	<0.5	0.5								
138.	"	14.180-5										
		Sawyer	0.7	<0.5								
139.	"	14.180-6										
		Sawyer	<0.5	<0.5								

SPECIAL SAMPLES

No.	Date	Identi.	$\mu\text{g/g}$									
			Al	Fe	Na	Li	K	C	Ag	Pt	Ge	
140.	11/18/83	QA30X X mined	10.1	<0.5	0.6	2.7	<0.5	<0.5	<0.5	<0.5		
141.	"	QA30X Z mined	<0.5	<0.5	0.5	<0.5	<0.5	<0.5	<0.5	<0.5		

TABLE II
Af Contract F-19628-81-C-0072
LITHIUM NIOBATE

Ident.	$\mu\text{g/g}$				
	Si	Mg	Fe	Ni	Al
LNB-1	235.	8.	1360.	320.	>2000.
3LN-6	102.	>1000.	1190.	275.	>2000.
UCN-2-4	64.	4.	1030.	230.	>2000.

Table III
AF Contract F-19628-81-C-0072
Hi Purity Sodium Carbonate

No.	'82 Date	Identi.	$\mu\text{g/g}$				
			Al	Fe	Li	Mg	Ca
1.	3/15	Unident.	2.	2.	0.8		
2.	4/29	Stauf. L	4.4	0.5	<0.5	16.	21.
3.	4/29	Stauf. D	2.	0.9	<0.5	20.	107.
4.	4/29	Baker 126325	<0.5	0.5	<0.5	2.	40.
5.	4/29	Allied Gr 1285	4.8	2.	<0.5	25.	51.
6.	4/29	Allied S 112	<0.5	5.1	<0.5	9.	107.
7.	4/29	CC #6	2.7	<0.5	0.6	16.	92.
8.	4/29	0.83 Molar	0.8	<0.5	280.	1.	3.3
9.	5/5	Stauf. L	1.4				
10.	5/5	Stauf. D	0.8				
11.	5/5	Baker 126325	0.6				
12.	5/5	Allied Gr 1285	4.3				
13.	5/5	Allied S 112(D)	0.5				
14.	5/5	CC #1382	2.0				
15.	5/5	Allied S (109)	1.7				
16.	5/5	Baker 038104	0.5				
17.	5/5	0.81 Molar	<0.5				
18.	5/5	Mal. 7528	<0.5	0.9	<0.5	13.	5.2

Table IV

AF Contract F-19628-81-C-0072

'82 Date	Identification	Al	Fe	Na	Li	K	Ca	Mg	Mn	P	Ti
		µg/g									
11-3	103H	46.	<0.5	3.	9.	1.	<0.5	<0.5			
"	113T	7.	<0.5	15.	2.	5.	<0.5	<0.5			
"	015JB	26.	<0.5	<0.5	5.	7.	<0.5	<0.5			
"	KAY	17.	<0.5	11.	0.6	2.	<0.5	<0.5			
"	COM	51.	<0.5	5.	3.	6.	<0.5	<0.5			
"	E8-51	19.	8.	625.	7.	<0.5	<0.5	<0.5			
"	E8-51 Supply After	32.	15.	1240.	3.	<0.5	<0.5	<0.5			
"	E8-51 " " Before	14.	1.	3.	0.5	<0.5	<0.5	<0.5			
"	14.5 Supply After	23.	6.	1890.	3.	<0.5	<0.5	<0.5			
"	13.191 " " Before	6.	<0.5	0.5	0.5	<0.5	<0.5	<0.5			
12-16	14.53-1	0.9	<0.5	1.4	<0.5	<0.5	1.8	0.6			
"	14.53-2	1.1	<0.5	0.8	0.5	<0.5	<0.5	<0.5			
"	14.53-3	7.0	<0.5	23.	2.2	<0.5	<0.5	<0.5			
"	14.53-4	1.0	<0.5	0.6	<0.5	<0.5	<0.5	<0.5			
"	14.56-1	3.0	33.	22.	2.3	<0.5	<0.5	<0.5			
"	14.56-2	27.	<0.5	25.	7.3	<0.5	<0.5	<0.5			
"	14.56-3	12.	0.7	4.9	3.6	<0.5	<0.5	<0.5			
"	F39B	880.	-	-	-	-	730.	919.	213.	<100.	760.
"	F40G	400.	-	-	-	-	0.5	203.	37.	<100.	78.
"	J15G	787.	-	-	-	-	3.5%	1.3%	1.3%	220.	278.

Table V
AF Contract F-19628-81-C-0072

Sample	$\mu\text{g/g}$					
	Al	Ca	Mg	Mn	P	Ti
F26 Bottom	780.	10800.	2020.	1160.	100.	690.
H38 Upper Wall	2590.	1480.	1990.	1650.	100.	505.
In-Ga Mix						
					94.7%	5.2%

TABLE VI
Indium Phosphide Samples
 $\mu\text{g/g}$

Sample	Fe	Ge	Sn
8-29-83			
1	ND	ND	35.
2	ND	ND	53.
3	ND	ND	63.
4	ND	ND	50.
5	ND	ND	61.
6	ND	ND	76.
7	ND	ND	640.
8	ND	ND	7800.
A	ND	2500.	ND
B	ND	2000.	ND
C	ND	126.	ND
D	580.	50.	ND
9-20-83			
Special (Ahern)	2.8		

Table VII
AF Contract F-19628-81-C-0072
Indium and Phosphorous Samples

Sample Identification	Al	Fe	Zn	Sn	Ga	Mg
Indium Corp. Indium	<1.	<1.	<0.5	<2.	<30.	<0.5
MPC Indium	<1.	<1.	<0.5	<2.	<30.	<0.5
MPC Red P form II	34.	<1.	<0.5	<2.	<30.	<0.5
MPC Red P (used)	<1.	<1.	<0.5	<2.	<30.	<0.5
Red P form II fresh	38.	17.	1.0	3.	<30.	<0.5
Red P form II spent	110.	1090.	4.	31.	<30.	1.8
InP LV-90	<1.	2.	<0.5	26.	<30.	<0.5

Appendix I

Analysis of Quartz for Impurities

Application: This method is applicable to the determination of aluminum, iron, sodium, lithium, potassium and silver. Two grams of sample are required for duplicate analyses.

Summary: A sample is dissolved in teflon in a Parr bomb using ultra-pure HF acid. The silica in the sample is then volatilized in platinum. The impurity containing residue is put into solution using ultra-pure HNO₃ acid. The analysis is completed using Atomic Absorption.

Apparatus:

1. Perkin-Elmer 5000 AA with graphite furnace capability.
2. Parr 4745 acid digestion bomb.
3. Platinum crucibles.
4. Volumetric flasks-10 ml.
5. Furnace or oven capable of 130 C.

Reagents and Standards:

1. Ultra-pure HF acid.
2. Ultra-pure HNO₃ acid.
3. Ultra-pure H₂SO₄ acid.
4. Ultra-pure acetone.
5. Demineralized water.
6. AA standards for the desired elements.

Analysis:

1. The quartz ingot is washed well with acetone and water to remove any sealing wax used in sawing.
2. The ingot is then wrapped in several layers of kleenex and broken into shards with a hammer.
3. The shards are then collected in a cleaned pyrex beaker and washed repeatedly with acetone, HCl, HNO₃ and demineralized water. They are finally washed with several changes of demineralized water. The shards are then dried carefully.
4. A sample is weighed from these cleaned and dried shards and placed in a similarly cleaned teflon cup for the Parr 4745 acid digestion bomb. The shards should be small.
5. Five ml. (for a 1 gm sample) of ultra-pure HF is added to the teflon cup and the bomb is carefully assembled.

6. The bomb is then placed in an oven and heated at 130 C overnight.. This should completely dissolve the sample but additional time may be needed if the shards are very large.
7. The bomb is then removed from the oven and allowed to cool.
8. The bomb is then carefully opened (wear rubber gloves and safety glasses) and the teflon cup removed. The cup is opened carefully to avoid spilling the contents and washed carefully into a clean platinum crucible.
9. Add 2 drops of ultra-pure H_2SO_4 and carefully heat the crucible to evaporate the HF and thus volatilized the silica as silicon tetrafluoride.
10. The residue remaining in the crucible (containing the impurity elements) is put back into solution by adding 3 drops of ultra-pure HNO_3 and a small amount of demineralized water. Heat gently but do not evaporate.
11. The contents of the crucible are very carefully transferred to a cleaned 10 ml volumetric flask and made to volume with demineralized water.
12. The analysis is completed using Atomic Absorption and the graphite furnace if necessary. Use carefully prepared standards and blanks.

Note: All glassware must be carefully cleaned before use. Do not get anything from the outside of the crucibles in the solution. Use rubber or plastic gloves and eye shields when handling HF and other acids.

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