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PREPARATION OF SYNTHETIC STANDARDS OF KNOWN  
CHEMICAL COMPOSITION FOR USE IN THE ANALYSIS  
OF COAL ASH BY X-RAY FLUORESCENCE

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INTRODUCTION

To carry out quantitative chemical analysis of coal ash and related products by x-ray fluorescence, standard reference materials, with a similar chemical composition to that of coal and related materials is essential. As such standard reference materials are not available, a choice of using coal ash analysed by chemical methods, or preparing synthetic standards is available. Coal ash analysed by chemical methods was considered too time consuming, as it would necessitate the collection of a large number of samples with a wide range of composition, which would have to be analysed chemically, synthetic standards could on the other hand be prepared from reagent grade chemicals with relative ease. The later choice was elected.

DETERMINATION OF THE PROPOSED COMPOSITION OF  
SYNTHETIC COAL ASH STANDARDS

It was decided that the synthetic ashes should simulate as closely as possible the chemical composition of ash of Canadian coals. The ten major elements in coal ash are listed in Table I.

TABLE I

Ten Major Elements in Coal Ash

Silicon	(Si)	Silica	(SiO <sub>2</sub> )
Aluminum	(Al)	Alumina	(Al <sub>2</sub> O <sub>3</sub> )
Iron	(Fe)	Iron Oxide	(Fe <sub>2</sub> O <sub>3</sub> )
Titanium	(Ti)	Titanium Dioxide	(TiO <sub>2</sub> )
Phosphorous	(P)	Phosphorus Pentoxide	(P <sub>2</sub> O <sub>5</sub> )
Calcium	(Ca)	Calcium Oxide	(CaO)
Magnesium	(Mg)	Magnesium Oxide	(MgO)
Sulphur	(S)	Sulphur Trioxide	(SO <sub>3</sub> )
Sodium	(Na)	Sodium Oxide	(Na <sub>2</sub> O)
Potassium	(K)	Potassium Oxide	(K <sub>2</sub> O)

To determine the range of composition required for the synthetic standards a study was made of "Chemical Analysis of the Ash of Canadian Coals"(1) which tabulates the per cent composition of the ten major constituents as oxides. From this study the range of composition was established. The ten oxides listed in Table I above make-up approximately 99 per cent of most coal ashes studied. Silica (SiO<sub>2</sub>) covers the widest

range. A table was constructed using 20 different ranges of silica from 0 to 100 per cent in increments of 5 per cent. The per cent composition of each of the other nine constituents was then entered on the table according to the per cent silica in the ash.

Following construction of this table it was noted that if the iron ( $\text{Fe}_2\text{O}_3$ ) content of two ashes was approximately the same, that the Calcium ( $\text{CaO}$ ) contents were also similar. On further examination this was confirmed regardless of the geographical source of the sample. Based on these observations a new table was constructed in which ashes with similar  $\text{Fe}_2\text{O}_3$  and  $\text{CaO}$  were grouped together, resulting in 19 different groups. The percentage composition of all ashes in each group were then averaged to give a mean representative value. See Table II.

Following construction of the table an attempt was made to prepare synthetic ashes with similar compositions.

#### EXPERIMENTAL

##### Preparation of Pure Chemicals for Use in the Preparation of Synthetic Coal Ash Standards

The chemicals used to prepare the synthetic coal ash standards and the oxide of the element which they supply are listed in Table III.

Primary standard chemicals used in the gravimetric analysis of the element of interest were employed wherever possible. All chemicals were ground to pass a No. 200 (.074 mm) sieve before ignition or drying, and stored under the conditions listed in Table III. As indicated in the table, the source of silica ( $\text{SiO}_2$ ) for the synthetic standards was silica gel ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), (FISCHER S-679). Since silica gel contains a variable

TABLE II

Per Cent Composition of Nineteen Representative Coal Ashes

GROUP NO.	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	S <sub>2</sub> O <sub>3</sub>	TOTAL
1	70.00	24.50	0.80	1.60	0.53	0.70	0.40	0.00	0.70	0.30	99.53
2	64.50	24.00	1.65	1.00	0.75	4.20	1.10	1.09	0.97	0.00	99.26
3	65.00	24.00	4.95	0.85	0.55	1.40	1.05	0.98	1.53	0.50	100.81
4	57.70	28.60	4.08	1.11	0.97	3.30	0.73	1.15	1.22	1.19	100.05
5	54.50	31.00	7.32	0.85	0.84	1.60	1.03	0.45	1.16	1.26	100.01
6	47.00	22.00	5.11	0.60	0.90	1.18	1.80	2.30	1.10	7.40	100.00
7	43.00	37.00	1.00	3.00	5.30	0.40	0.20	0.10	7.00	3.14	100.14
8	42.67	30.66	7.13	1.20	2.07	6.66	1.00	1.30	0.76	6.53	99.98
9	41.60	21.70	25.00	0.67	0.53	3.56	1.27	0.63	2.22	2.78	99.96
10	32.00	22.00	12.40	0.50	0.40	15.50	2.10	2.10	0.80	12.10	100.00
11	31.67	20.83	5.97	0.43	0.93	17.83	2.73	5.63	0.67	13.30	99.99
12	29.00	19.25	40.00	0.60	0.50	3.80	1.15	0.70	1.30	3.70	100.00
13	25.00	18.00	19.50	0.40	0.55	16.00	2.95	2.00	0.65	14.95	100.00
14	23.00	16.00	6.00	0.40	0.70	25.00	5.20	8.20	0.50	15.00	100.00
15	22.00	17.00	50.00	0.50	0.40	3.40	0.90	0.60	0.90	4.30	100.00
16	17.00	14.00	58.00	0.40	0.60	3.10	0.90	0.70	0.70	4.60	100.00
17	16.00	14.00	8.60	0.40	0.50	31.00	5.50	6.40	0.50	17.00	100.00
18	15.00	10.00	70.00	0.05	0.50	3.00	0.10	0.70	0.33	0.90	100.58
19	15.00	10.00	26.00	0.30	0.20	20.00	3.20	0.90	0.80	23.60	100.00

TABLE III

## Chemicals Used for Preparation of Synthetic Coal Ashes

CHEMICAL USED	OXIDES SUPPLIED	IGNITED OR DRIED AT *under oxidizing conditions	STORAGE CONDITIONS
Silica - GEL $\text{SiO}_2 \cdot n\text{H}_2\text{O}$	$\text{SiO}_2$	$1100^\circ\text{C}$ for at least 12 hrs.*	in desiccator over $\text{MgClO}_4$
Alundum $\text{Al}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$1100^\circ\text{C}$ for at least 12 hrs.*	in desiccator over $\text{MgClO}_4$
Ferric Oxide $\text{Fe}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	$750^\circ\text{C}$ for at least 12 hrs.*	in tightly sealed jar
Titanium Dioxide $\text{TiO}_2$	$\text{TiO}_2$	$750^\circ\text{C}$ for at least 12 hrs.*	in desiccator over $\text{MgClO}_4$
Calcium Carbonate $\text{CaCO}_3$	$\text{CaO}$	$105^\circ\text{C}$ for at least 12 hrs.	in desiccator over $\text{MgClO}_4$
Calcium Sulphate Dihydrate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	$\text{CaO}$ $\text{SO}_3$	$750^\circ\text{C}$ for at least 12 hrs.*	in desiccator over $\text{MgClO}_4$
Magnesium Oxide $\text{MgO}$	$\text{MgO}$	$1100^\circ\text{C}$ for at least 12 hrs.*	in desiccator over $\text{MgClO}_4$
Sodium Sulphate $\text{Na}_2\text{SO}_4$	$\text{Na}_2\text{O}$	$105^\circ\text{C}$ for at least 12 hrs.	in desiccator over $\text{MgClO}_4$
Potassium Sulphate $\text{K}_2\text{SO}_4$	$\text{K}_2\text{O}$ $\text{SO}_3$	$105^\circ\text{C}$ for at least 12 hrs.	in desiccator over $\text{MgClO}_4$
Monopotassium Phosphate $\text{KH}_2\text{PO}_4$	$\text{K}_2\text{O}$ $\text{P}_2\text{O}_5$	$105^\circ\text{C}$ for at least 12 hrs.	in desiccator over $\text{MgClO}_4$



amount of adsorbed water special treatment was necessary to convert the silica gel to a stable form of silica. This was accomplished by heating the unground silica gel (100-200 mesh) to 1100°C in a clean muffle furnace for 12 hours. The ignited silica was then cooled in a desiccator over magnesium perchlorate, and ground to pass a No. 200 (.074 mm) sieve, and re-ignited in a muffle at 1100°C to constant weight as described in Table III. No special precautions other than those listed in the table were taken with the other chemicals.

#### Preparation of the Synthetic Coal Ash Standards from the Prepared "Pure" Chemicals

Each synthetic coal ash was prepared by mixing the "pure" chemicals according to the weight proportions given in Table IV. Each chemical was weighed on an analytical balance to the nearest 0.001 grams and transferred directly to a stainless steel sieve receiver. Four rubber stoppers were placed in the receiver with the chemicals, and a sieve cover placed on the receiver. The contents of the receiver were mixed by shaking on a Gilson Sieve Tester (Model SS-15) for fifteen minutes. Each prepared synthetic ash was then transferred to a clean glass bottle and stored in a desiccator over magnesium perchlorate.

For all 19 synthetic ash standards prepared according to the weight proportions given in Table IV the total weight contributed by the ten major oxides was 100.00 grams. However, it is evident from Table IV that the "actual" total weight of all 19 synthetic ashes was greater than 100.00 grams.

Monopotassium phosphate ( $\text{KH}_2\text{PO}_4$ ) was used as a source of potassium oxide ( $\text{K}_2\text{O}$ ) and phosphorous pentoxide ( $\text{P}_2\text{O}_5$ ), and calcium carbonate ( $\text{CaCO}_3$ )

TABLE IV

Weight Proportions of Chemicals Required for Preparation of Synthetic Ashes

SYNTHETIC STANDARD	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	CaCO <sub>3</sub>	CaSO <sub>4</sub>	MgO	Na <sub>2</sub> SO <sub>4</sub>	K <sub>2</sub> SO <sub>4</sub>	KH <sub>2</sub> PO <sub>4</sub>	OXIDE TOTAL
1	70.000	24.500	0.800	1.600	1.250	0.000	0.800	0.070	0.648	1.014	100.682
2	63.970	22.000	1.650	1.000	7.500	0.000	1.100	3.910	0.924	1.436	103.490
3	64.500	22.600	4.950	0.850	2.500	0.000	1.050	1.604	2.110	1.054	101.218
4	58.000	27.480	4.000	1.100	5.893	0.000	0.700	2.634	1.304	1.724	102.835
5	54.500	31.000	7.300	0.850	2.750	0.136	1.260	0.820	1.184	1.628	101.428
6	47.000	22.000	5.100	0.600	16.010	6.884	1.800	5.038	1.118	1.724	107.274
7	43.000	36.910	1.000	3.000	0.714	0.000	0.200	0.230	6.456	10.150	101.660
8	42.350	31.000	7.100	1.200	6.139	7.432	1.000	3.068	0.000	3.926	103.215
9	41.500	22.000	25.000	0.650	5.796	0.616	1.300	1.374	3.398	1.054	102.688
10	32.000	22.000	12.400	0.500	16.696	15.181	2.100	4.810	1.000	0.766	107.453
11	31.500	21.000	5.950	0.400	23.982	10.728	2.700	11.680	1.118	1.724	110.782
12	29.000	19.300	40.000	0.600	4.380	3.269	1.150	1.603	1.794	0.958	102.054
13	25.000	18.000	19.500	0.400	13.359	20.677	2.950	4.397	0.677	1.053	106.013
14	23.000	16.000	6.000	0.400	38.913	7.796	5.200	17.794	0.860	1.340	117.303
15	22.000	17.000	50.000	0.500	2.325	5.093	0.900	1.374	1.157	0.766	101.115
16	17.000	14.000	58.000	0.280	1.398	5.914	0.900	1.397	0.727	1.149	100.765
17	16.000	14.000	8.600	0.400	44.380	14.918	5.500	14.290	0.609	0.958	119.655
18	15.000	10.000	70.000	0.050	5.357	0.000	0.100	1.023	0.000	0.958	102.488
19	15.000	10.000	26.000	0.300	8.357	37.188	3.200	2.061	1.235	0.383	103.724

was used to supply calcium oxide. The monopotassium phosphate also contributes water ( $H_2O$ ) and the calcium carbonate contributes carbon dioxide to each synthetic ash. Hence, the actual total weight of each synthetic ash was greater than 100.00 grams because of the weight contributed by the water of hydration of the monopotassium phosphate and the carbon dioxide from the calcium carbonate. However, it was decided that the carbon dioxide from the calcium carbonate and the water from the monopotassium phosphate in each synthetic ash would be lost on ignition of the ash at  $750^{\circ}C$  and as result each synthetic ash would attain the theoretical per cent composition given in Table V.

#### Analytical Procedures

Ten of the nineteen synthetic ashes were subjected to a series analyses. Analyses performed on the ashes were, loss on ignition at  $750^{\circ}C$ , mineral analyses (silicon, aluminum, iron, titanium, calcium and magnesium) by atomic absorption, sodium, and potassium by flame photometry, analysis of phosphorous by spectrophotometry, and analysis for sulphur by the LECO IR-33 sulphur determinator. An outline of the important features of each methods of analysis is discussed below.

##### 1. Loss on Ignition

3 to 5 grams of sample passing a number 200 sieve was ignited at  $750^{\circ}C$  to constant weight ( 0.001g). The loss in weight of the synthetic ash was used to calculate loss on ignition.

##### 2. Metal Oxide Analyses by Atomic Absorption

$0.2 \pm 0.0001$  grams of synthetic ash was weighed into a clean platinum crucible containing 0.5 g lithium tetraborate. The synthetic ash and the lithium tetraborate were well mixed, and then 0.5 grams of

TABLE V

Theoretical Per Cent Compositions of 19 Synthetic Coal Ashes After Ignition at 750°C

NO.	TOTAL	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>
1	100.00	70.00	24.50	0.80	1.60	0.53	0.70	0.80	0.30	0.70	0.34
2	100.00	63.97	22.00	1.65	1.00	0.75	4.20	1.10	1.72	1.00	2.61
3	100.00	64.50	22.60	4.95	0.85	0.55	1.40	1.05	0.70	1.50	1.90
4	100.00	58.00	27.48	4.00	1.10	0.90	3.30	0.70	1.15	1.30	2.08
5	100.00	54.50	31.00	7.30	0.85	0.85	1.60	1.26	0.36	1.20	1.09
6	100.00	47.00	22.00	5.10	0.60	0.90	11.80	1.80	2.20	1.20	7.40
7	100.00	43.00	36.91	1.00	3.00	5.30	0.40	0.20	0.10	7.00	3.10
8	100.00	42.35	31.00	7.10	1.20	2.05	6.50	1.00	1.34	1.36	6.10
9	100.00	41.50	22.00	25.00	0.65	0.55	3.50	1.30	0.60	2.20	2.70
10	100.00	32.00	22.00	12.40	0.50	0.40	15.60	2.10	2.10	0.80	12.10
11	100.00	31.50	21.00	5.95	0.40	0.90	17.85	2.70	5.10	1.20	13.40
12	100.00	29.00	19.30	40.00	0.60	0.50	3.80	1.15	0.70	1.30	3.65
13	100.00	25.00	18.00	19.50	0.40	0.55	16.00	2.95	1.92	0.73	14.95
14	100.02	23.00	16.00	6.00	0.40	0.70	25.00	5.20	7.83	0.93	14.96
15	100.00	22.00	17.00	50.00	0.50	0.40	3.40	0.90	0.60	0.90	4.30
16	100.00	17.00	14.00	58.00	0.28	0.60	3.22	0.90	0.61	0.79	4.60
17	100.00	16.00	14.00	8.60	0.40	0.50	30.97	5.50	6.29	0.66	17.08
18	100.00	15.00	10.00	70.00	0.05	0.50	3.00	0.10	0.45	0.33	0.57
19	100.00	15.00	10.00	26.00	0.30	0.20	19.93	3.20	0.91	0.80	23.66

additional lithium tetraborate was added to cover the mixture. The mixture was fused in a muffle at  $1000^{\circ}\text{C}$  for at least 15 minutes. After the fusion was complete, the cooled melt was taken up in 5% HCl and quantitatively transferred to a clean 400 ml. beaker. The resulting solution was transferred to a 200 ml. volumetric flask and diluted to the mark with 5% HCl. This solution was then analysed for six of the ten major oxides present in the synthetic ashes by atomic absorption techniques. The wave lengths and gas mixtures used to analyse for these six major oxides are given in Table VI.

### 3. Metal Oxide Analyses (by Flame Photometry)

The sodium and potassium oxides present in each synthetic standard were determined using a flame photometer. A 20 ml. aliquot of the solution used for the atomic, absorption analysis was transferred to a 100 ml. volumetric flask, containing 5 ml of a 600 ppm lithium solution. The solution in the flask was diluted to the mark with distilled water and mixed well. The sodium and potassium were then determined on a flame photometer calibrated with solutions containing known concentrations of sodium and potassium.

### 4. Determination of Phosphorous Pentoxide by Spectrophotometry

Phosphorous pentoxide in each of the synthetic ashes was determined by spectrophotometry. A 30 ml. aliquot of the solution used for atomic absorption analyses was transferred to a 100 ml. beaker. Exactly 10 ml. of a molybdivanadate solution (0.6 g  $\text{Na VO}_3$  in 200 ml. 1:1  $\text{HNO}_3$ , 380.0 grams  $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$  in 200 ml. water. mix and dilute to 1 l.) was added to the beaker. The solution was allowed to stand for 5 minutes and the absorbance read at 420 nm. By comparing the absorbance of the solution to the absorbance of a standard the per cent  $\text{P}_2\text{O}_5$  was determined.

TABLE VI

Wavelengths and Gas Mixtures Used for  
Metal Analyses by Atomic Absorption

METAL	OXIDE DETERMINED	WAVELENGTH nm	FUEL	OXIDIZING AGENT
Si	SiO <sub>2</sub>	2516	Acetylene	Nitrous oxide
Al	Al <sub>2</sub> O <sub>3</sub>	3092	Acetylene	Nitrous oxide
Fe	Fe <sub>2</sub> O <sub>3</sub>	2483	Acetylene	Air
Ti	TiO <sub>2</sub>	3643	Acetylene	Nitrous oxide
Ca	CaO	4227	Acetylene	Air
Mg	MgO	2851	Acetylene	Air

TABLE VII  
Results of Analyses of Ten Synthetic Coal Ashes

ELEMENTAL OXIDE	STANDARD											ALLOWABLE DIFFERENCE (ASTM)
	S-2	S-4	S-5	S-6	S-7	S-9	S-10	S-13	S-14	S-15		
SiO <sub>2</sub>	A	62.65	56.50	52.64	45.33	42.22	40.46	32.50	25.30	23.28	21.50	2.00
	T	63.97	58.00	54.50	47.00	43.00	41.50	32.00	25.00	23.00	22.00	
Al <sub>2</sub> O <sub>3</sub>	A	21.41	26.33	29.58	22.22	36.38	21.61	22.18	17.82	15.92	17.23	2.00
	T	22.00	27.48	31.00	22.00	36.91	22.00	22.00	18.00	16.00	17.00	
Fe <sub>2</sub> O <sub>3</sub>	A	1.51	3.69	7.40	5.45	0.67	24.33	12.36	19.00	5.71	50.05	0.70
	T	1.65	4.00	7.30	5.10	1.00	25.00	12.40	19.50	6.00	50.00	
TiO <sub>2</sub>	A	1.04	1.03	0.75	0.60	+3.40	0.69	0.47	0.42	0.47	0.53	0.25
	T	1.00	1.10	0.85	0.60	3.00	0.65	0.50	0.40	0.40	0.50	
P <sub>2</sub> O <sub>5</sub>	A	0.83	0.98	0.96	1.01	+5.09	*0.92	*0.70	*0.85	0.85	*1.42	0.15
	T	0.75	0.90	0.85	0.90	5.30	0.55	0.40	0.55	0.70	0.40	
CaO	A	4.40	3.38	1.85	11.98	0.44	3.66	15.64	15.62	24.86	3.48	0.40
	T	4.20	3.30	1.60	11.80	0.40	3.50	15.60	16.00	25.00	3.40	
MgO	A	1.05	0.68	1.18	1.80	0.17	1.25	2.03	2.61	+4.44	0.81	0.50
	T	1.10	0.70	1.26	1.80	0.20	1.30	2.10	2.95	5.20	0.90	
SO <sub>3</sub>	A	2.48	2.09	1.20	7.58	3.07	2.71	12.17	15.15	15.09	4.26	0.20
	T	2.61	2.08	1.19	7.40	3.10	2.70	12.10	14.95	14.96	4.30	
Na <sub>2</sub> O	A	1.51	1.18	0.54	2.19	0.22	0.69	2.12	1.85	+7.00	0.68	0.30
	T	1.72	1.15	0.36	2.20	0.10	0.60	2.10	1.92	7.83	0.60	
K <sub>2</sub> O	A	0.90	1.14	1.17	1.02	+6.14	1.94	0.74	0.68	0.85	0.77	0.30
	T	1.00	1.30	1.20	1.20	7.00	2.20	0.80	0.73	0.93	0.90	
TOTAL	A	97.71	96.90	97.16	99.09	97.21	98.08	100.85	99.25	98.40	100.66	
	T	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	
Loss on ignition	A	3.50	2.94	1.55	6.69	1.82	2.83	6.99	5.87	14.65	1.18	
	T	3.19	2.75	1.61	6.75	1.63	2.62	6.94	5.67	14.75	1.10	

NOTE: A - Analytical  
T - Theoretical

Determination of Sulphur using a Leco Induction Furnace and a Leco Sulphur Determinator

0.05 to 0.06 grams of synthetic ash was weighed into a ceramic crucible. A small amount of iron chip catalyst was added to the crucible, and sufficient vanadium pentoxide powder to completely cover the sample. A solid wafer of vanadium pentoxide was placed in the crucible to cover the mixture and prevent sample blow-out. The crucible was transferred to a Leco induction furnace which converts the sulphur in each sample to sulphur dioxide by high temperature combustion. The sulphur dioxide produced is detected using an infrared cell and read-out as per cent S on a digital voltmeter. The system is calibrated using ashes containing known percentages of S which has been previously determined by the ASTM Method D 1757.

RESULTS AND DISCUSSION

A list of the results obtained from the analyses of the ten synthetic ashes is given in Table VII. The results which exceed the ASTM limits of reproducibility as given in Table VII are either marked with a plus or an asterisk. The results marked with a plus indicate cases where the theoretical concentration of the elemental oxide in question was beyond the range of standards used in the analysis of the oxide. Hence the discrepancy between the analytical and theoretical values might be partly accounted for. The results marked with an asterisk indicate situations where systematic errors in the analytical techniques might have contributed to a difference between the theoretical and determined results, which exceeded ASTM limits of reproducibility. This situation occurred in the case of phosphorous pentoxide ( $P_2O_5$ ).



The presence of iron in an ash sample interferes with the determination of phosphorous pentoxide using spectrophotometry. It is pointed out in the literature that the absorbance values obtained in the determination of phosphorous are enhanced by the presence of iron (2) consequently, the calculated values for phosphorous pentoxide must be corrected for the quantity of iron oxide in the sample. This was done using the equation given below:

$$(1) \%P_{2O_5} \text{ actual} = \%P_{2O_5} \text{ apparent} - 0.0025\% Fe_2O_3$$

Using equation 1 above the results given in Table VII were obtained for phosphorous pentoxide.

For any synthetic ash containing less than 10% iron oxide as  $Fe_2O_3$ , the analytical value for  $P_2O_5$  after correction for the amount of  $Fe_2O_3$  present using Equation 1, agreed with the theoretical value for  $P_2O_5$  within ASTM limits with one exception. The exception occurred in synthetic standard 7. In this synthetic standard the theoretical concentration of phosphorous pentoxide was beyond the range of the standard used in the analysis of phosphorous pentoxide. For any synthetic ash containing more than 10% iron oxide as  $Fe_2O_3$ , the analytical value for  $P_2O_5$  after correction for the amount of  $Fe_2O_3$  present using Equation 1, did not agree with the theoretical value for  $P_2O_5$  within ASTM limits. As the percent iron oxide as  $Fe_2O_3$  in the sample increased from 12% to 50% the error in the  $\%P_2O_5$  increased from 0.27% to 1.02%.

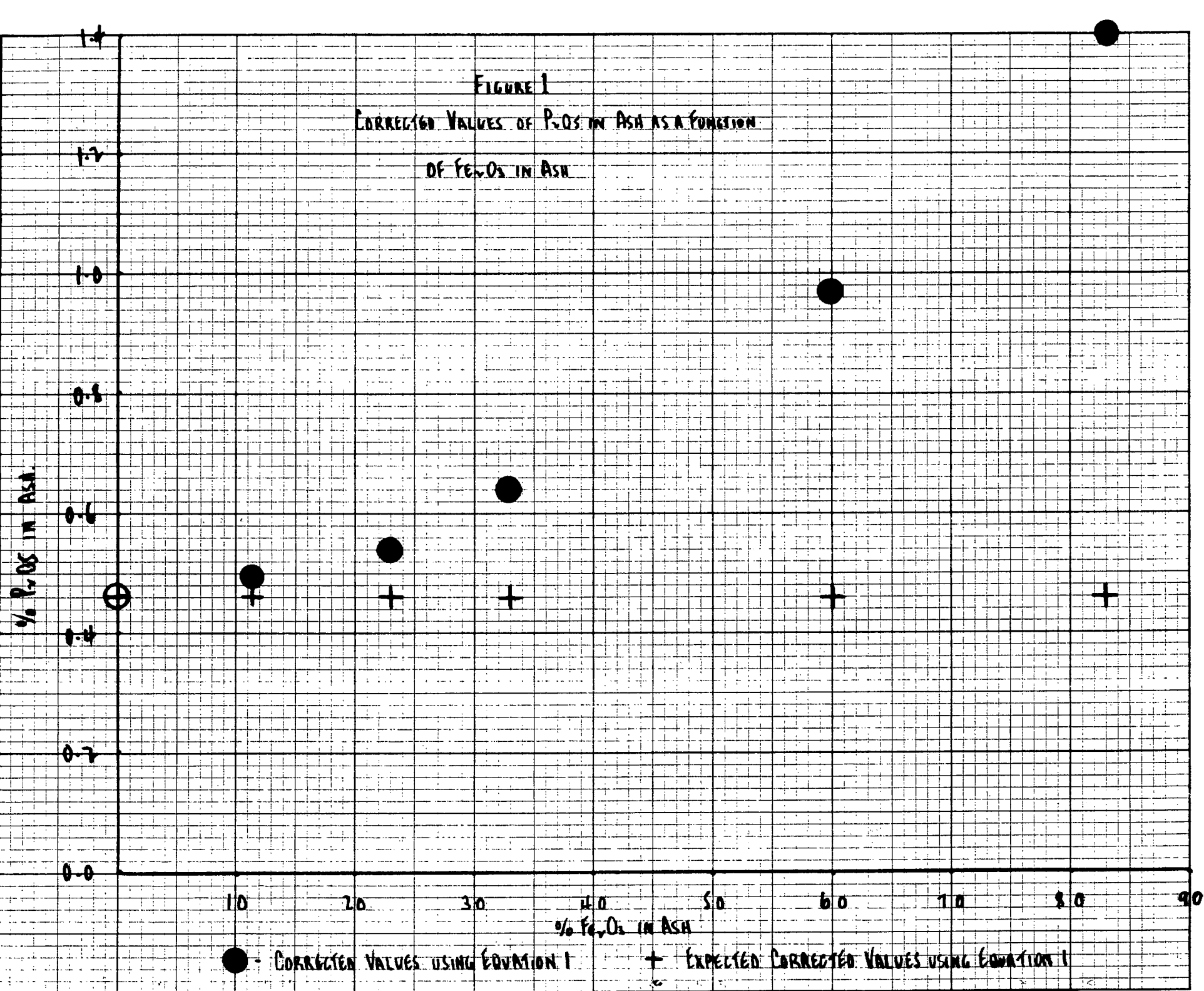
Figure 1 illustrates that for an ash containing 0.43%  $P_2O_5$  and amounts of  $Fe_2O_3$  varying from zero to 83.00% that Equation 1 does not adequately correct for the amount of  $Fe_2O_3$  in the ash, the results plotted

in Figure 1 were obtained by preparing a solution containing phosphorous equivalent to 0.43%  $P_2O_5$  in an ash. Amounts of iron oxide as  $Fe_2O_3$  equivalent to zero% to 83% in an ash were added to the phosphorous solution. The absorbance of each solution was determined as described in the analytical procedures. From the absorbance of each solution the apparent % $P_2O_5$  was determined. The apparent % $P_2O_5$  was then corrected for % $Fe_2O_3$  in the solution to give the analytically corrected value for % $P_2O_5$ . In each case the analytically corrected value for % $P_2O_5$  using Equation 1 should have been 0.43% as indicated by the X's in Figure 1. The actual analytically corrected values obtained using Equation 1 are indicated by the O's in Figure 1. It is evident from Figure 1 that the difference between the actual corrected analytical value for  $P_2O_5$  using equation 1 and the expected corrected analytical value of 0.43% increases as the % $Fe_2O_3$  increases.

#### CONCLUSIONS

1. Synthetic ashes containing given amounts of elemental oxides can be prepared from pure chemicals.
2. The actual value of phosphorous pentoxide in an ash is not given by the Equation:

$$\%P_2O_5 \text{ actual} = \%P_2O_5 \text{ apparent} - (0.0025\% Fe_2O_3)$$



REFERENCES

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2. ASTM Document D29.05. Proposed method for the correction of  $P_2O_5$  due to the presence of  $Fe_2O_3$ .