ELSEVIER

Thermochimica Acta 259 (1995) 235–249

thermochimica
acta

Determination of volatile material and ash content in polymeric sulfur by thermogravimetric analysis

C. Rial *, J.M. Jiménez-Mateos

REPSOL, Centro de Investigación, C/ Embajadores 183, 28045-Madrid, Spain

Received 13 October 1994; accepted 24 January 1995

Abstract

The volatile matter and ash content in polymeric sulfur are two important parameters for the characterization and process control of this material. These parameters are traditionally determined by two different standard test methods that require a long time and large amounts of sample. In the present work, a test method based on a single thermogravimetric analysis for the simultaneous determination of volatile material and ash content in a sulfur specimen is proposed as an alternative to the standard methods.

Keywords: Ash content; Polymeric sulfur; TG; TGA; Volatile matter

---

List of symbols

A	percent ash content in a sulfur specimen
$\langle A \rangle$	arithmetic average of the percent ash content in a sulfur specimen
$\langle A_{est} \rangle$	arithmetic average of the estimated percent ash content in a sulfur specimen (estimated from the average weight of the residue at 800°C minus the systematic error, $(\langle W_{800^\circ C} \rangle - \langle SE \rangle)$)
df	degrees of freedom
F	statistic used to compare two variances
g	statistic used to compare more than two variances (Cochran test)
M_o	original specimen mass
M_r	final specimen mass or ashes
s	standard deviation

---

* Corresponding author.

SSDI 0040-6031(95)02281-3

s_p	pooled standard deviation
s_{SE}	standard deviation of the systematic error
s^2	variance
$\langle \mathrm{SE} \rangle$	systematic error (%) (arithmetic average of the calculated differences ($W_{800^\circ\mathrm{C}} - A$) for all the determinations using the TGIII test)
t	student's statistic used for the comparison of two averages
$W_{800^\circ\mathrm{C}}$	weight of the residue (%) at 800°C
$\langle W_{800^\circ\mathrm{C}} \rangle$	arithmetic average of the weight of the residue at 800°C for each sample
ΔW	difference between the weight of the residue at 800°C and the ash content measured at 30°C for each determination ($W_{800^\circ\mathrm{C}} - A$)
$\langle \Delta W \rangle$	arithmetic average of the differences ($W_{800^\circ\mathrm{C}} - A$) for each sample
α	confidence level (0.05 in this work)

Methods

ACD	traditional determination of the ash content, based on Ref. [2]
VMD	traditional determination of the volatile matter content, based on Ref. [1]
TGI	thermogravimetric method, modification I
TGII	thermogravimetric method, modification II
TGIII	thermogravimetric method, modification III

1. Introduction

The characterization of sulfur is of great importance to the chemical industry because of the wide range of applications of this material. Regarding this point, the determination of the volatile matter and the ash content in sulfur is essential for many of its uses. Standard test methods suitable for measuring these parameters have been developed and issued by the ASTM [1,2]. The volatile matter content of polymeric sulfur (mainly water ("moisture") and low boiling point organic components) is currently determined from the loss of mass after heating the specimen for 2 h at 70°C [1]. The ash content of the specimen is usually obtained from the weight of the residue after ashing the sample at 600–800°C [2]. These traditional methods are time-consuming and demand large amounts of sample.

Thermogravimetric analysis (TGA) is a powerful tool for studying the evolution of the weight of a material with temperature and/or time. Thus, the determinations of the volatile matter and the ash content in a sulfur specimen can be included in the field of applications of this technique. The aim of the present work is to present a new method for measuring the volatile matter and the ash content in polymeric sulfur from a single TGA experiment. The results obtained by the TGA method do not differ significantly from those yielded by the traditional tests, being as precise as these. This fact, together with the simplicity of the procedure and the small size of sample required, make this new method suitable for quality control. Besides these advantages, the TGA method allows the evolution of the weight of the specimen with temperature to be recorded over the whole operation. It is then possible to determine the weight of the sample at any point of the experiment and to compare the measured curves of different samples to find differences in their thermal behavior.

2. Experimental

First of all, the volatile material and the ash content in three representative samples of polymeric sulfur (named S1, S2 and S3) were determined using the traditional methods. Then, for the adaptation of TGA, the experimental conditions used in the traditional tests were simulated in the thermogravimetric analyzer. These conditions were progressively modified in order to reduce the measuring time, the results being tested after each modification to avoid significant changes in the precision of the determinations.

Thermogravimetric analyses were performed on a Perkin Elmer TGA 7 thermogravimetric analyzer, equipped with a high-temperature furnace (balance sensitivity 0.01 mg, temperature precision ±5°C). Analysis of the data was carried out using the Perkin Elmer 7 Series / UNIX Software.

The traditional methods and the different TG tests are briefly described below.

2.1. *Traditional methods*

2.1.1. *Determination of volatile matter (VMD) (based on Ref. [1])*

A sulfur specimen of approximately 5 g is weighed and heated in an oven at 70°C for 2 h. Then, the sample is cooled in a desiccator for 1 h and weighed again. The volatile matter content is calculated using the equation

$$V(\%) = [(M_0) - (M_r)/M_0] \times 100$$

2.1.2. *Determination of ash content (ACD) (based on Ref. [2])*

A sulfur specimen of approximately 10 g is weighed into a previously ignited and weighed porcelain crucible. The sample is heated to 500°C and burned off before heating to 800°C for 2 h. Then, the crucible is cooled in a desiccator and weighed. The percent ash is calculated as

$$A(\%) = (M_r/M_0) \times 100$$

2.2. *Thermogravimetric methods*

All the thermogravimetric experiments were performed under flowing dry synthetic air ($90\,\mathrm{cc\,min^{-1}}$, $60\,\mathrm{cc\,min^{-1}}$ balance purge, $30\,\mathrm{cc\,min^{-1}}$ sample purge). Polymeric sulfur specimens of about 120 mg were weighed in the platinum crucible of the analyzer.

2.2.1. *Thermogravimetric method I (TGI)*

This test is a simulation in the thermogravimetric analyzer of the traditional method for determining the ash content in a sulfur sample (ACD). The sample is heated at a rate of 10°C min^{-1} to 500°C, and maintained at this temperature for 30 min to burn it off. Then, the temperature is increased to 800°C at a rate of 20°C min^{-1} and kept constant for 70 min. The resulting ash is weighed at this temperature ($W_{800°C}$(%)) and once again at 30°C (A(%)), after cooling the crucible in the thermogravimetric analyzer.

2.2.2. *Thermogravimetric method II (TGII)*

The volatile matter and the ash content in a sulfur specimen are determined by this test, combining the VMD and the ACD methods in a single thermogravimetric run. The specimen is heated to 70°C at a rate of 4°C min^{-1} and maintained at this temperature for 2 h. The loss of mass observed during this isothermal stage corresponds to the volatile matter content of the sample (V(%)) and can be directly determined on the TG curve. Next, the sample is heated to 500°C at a rate of 20°C min^{-1} and the temperature is kept constant for 15 min to allow the sulfur to burn off. Then, the temperature is increased to 800°C at a rate of 30°C min^{-1} and an isothermal stage is recorded for 45 min. After that, the weight of the residue at 800°C ($W_{800°C}$(%)) is determined on the thermogram. Finally, the assembly is allowed to cool to 30°C and the residue is weighed again (A(%)).

2.2.3. *Thermogravimetric method III (TGIII)*

In this TG test for the determination of the volatile matter and the ash content in a polymeric sulfur specimen, the measuring time has been reduced relative to the TGII method without changing significantly the precision of the results. A typical thermogravimetric run using this method is shown in Fig. 1. The sample is heated to 70°C at a rate of 4°C min^{-1} and the loss of mass corresponding to the volatile matter is determined on the TG curve after an isothermal stage of 2 h (V(%)) (Fig. 2). Next, the specimen is heated to 800°C at a rate of 30°C min^{-1}, and calcinated at this temperature for 45 min. At the end of this stage, the weight of the residue at 800°C is measured on the thermogram ($W_{800°C}$(%)). Finally, the assembly is allowed to cool and the weight of the residue at 30°C is determined (A(%)) (Fig. 3).

3. Results and discussion

3.1. *Results*

As previously mentioned in the experimental section, the volatile matter (V(%)) and the ash content (A(%)) in three polymeric sulfur samples (S1, S2 and S3) were first determined using the traditional methods (VMD and ACD, respectively). The results of the determinations, repeated four times on each sample, are shown in Tables 1 and 2.

For the development of the TG method, the steps detailed below were followed.

First, A(%) was determined by the TGI test, simulating the conditions of the ACD method in the thermobalance. The measurements were repeated four times on each sample (Table 2) to test the average value and the precision of the determination for significant differences compared with the values obtained by the traditional method (Table 4).

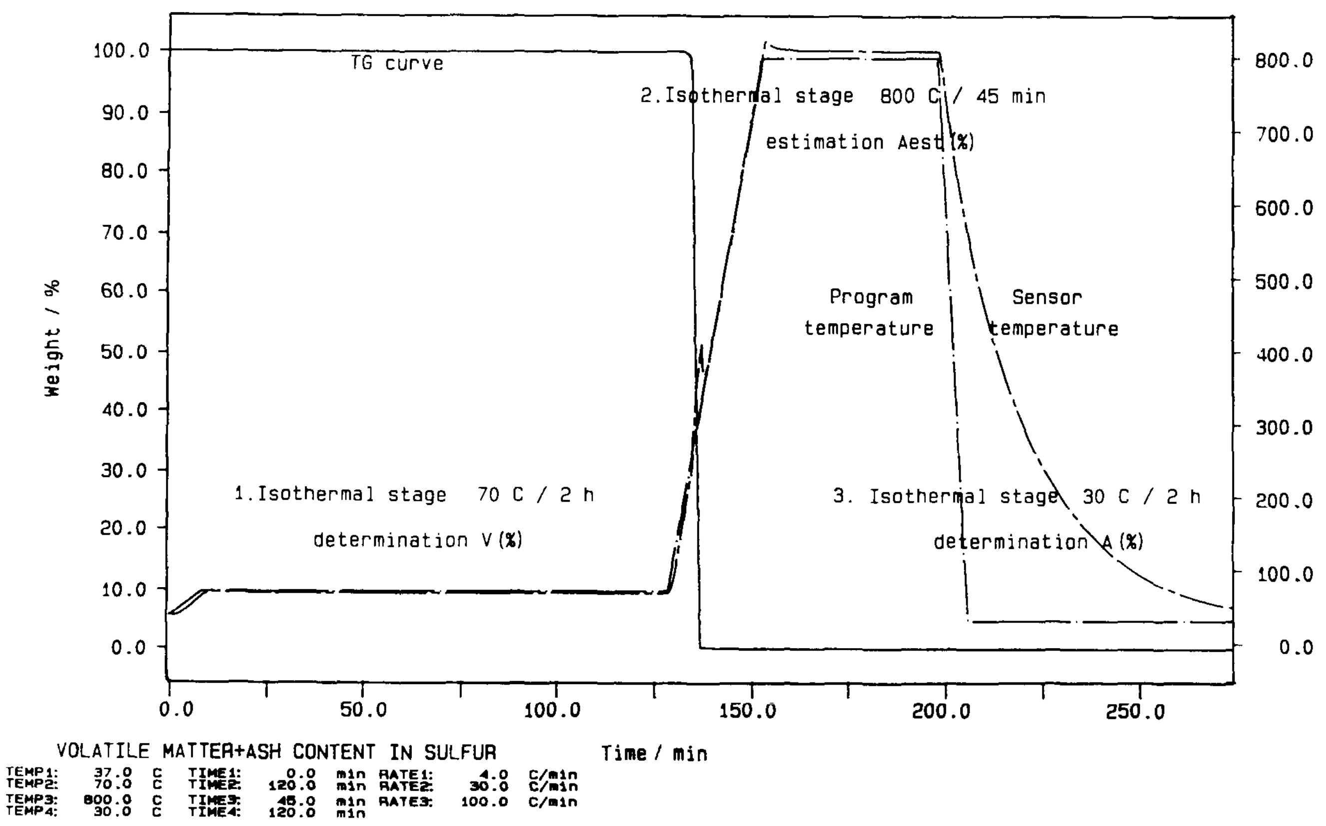

Fig. 1. Typical TGA curve using the TGIII method for the determination of volatile material (V(%)) and ash content (A(%)) in a sulfur specimen. The program and the sensor temperature curves are also shown.

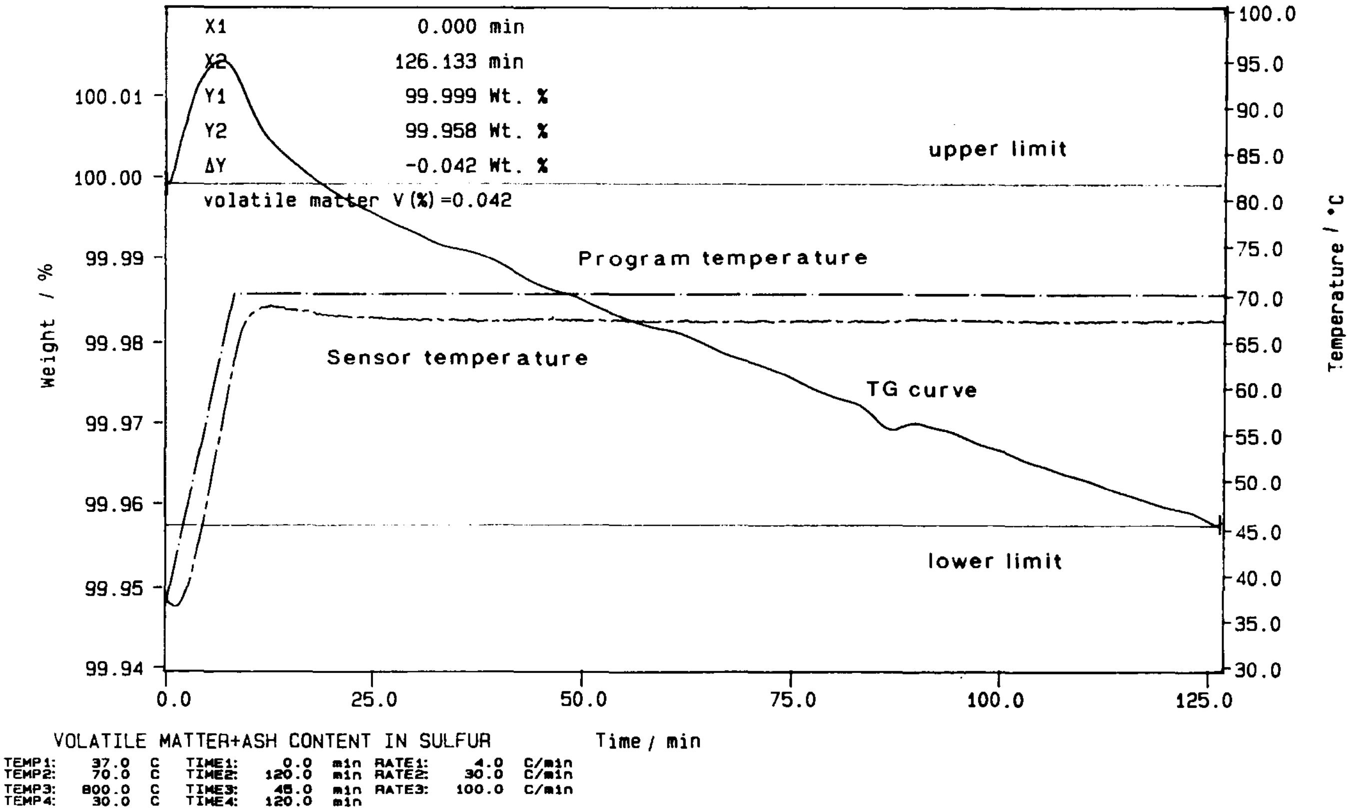

Fig. 2. Determination of the volatile matter content in a sulfur sample using the TGIII method. V(%) is directly determined on the measured curve from the observed loss of mass after an isothermal stage at 70°C for 2 h. The upper and the lower limits for calculation, together with the program and the sensor temperature curves are included.

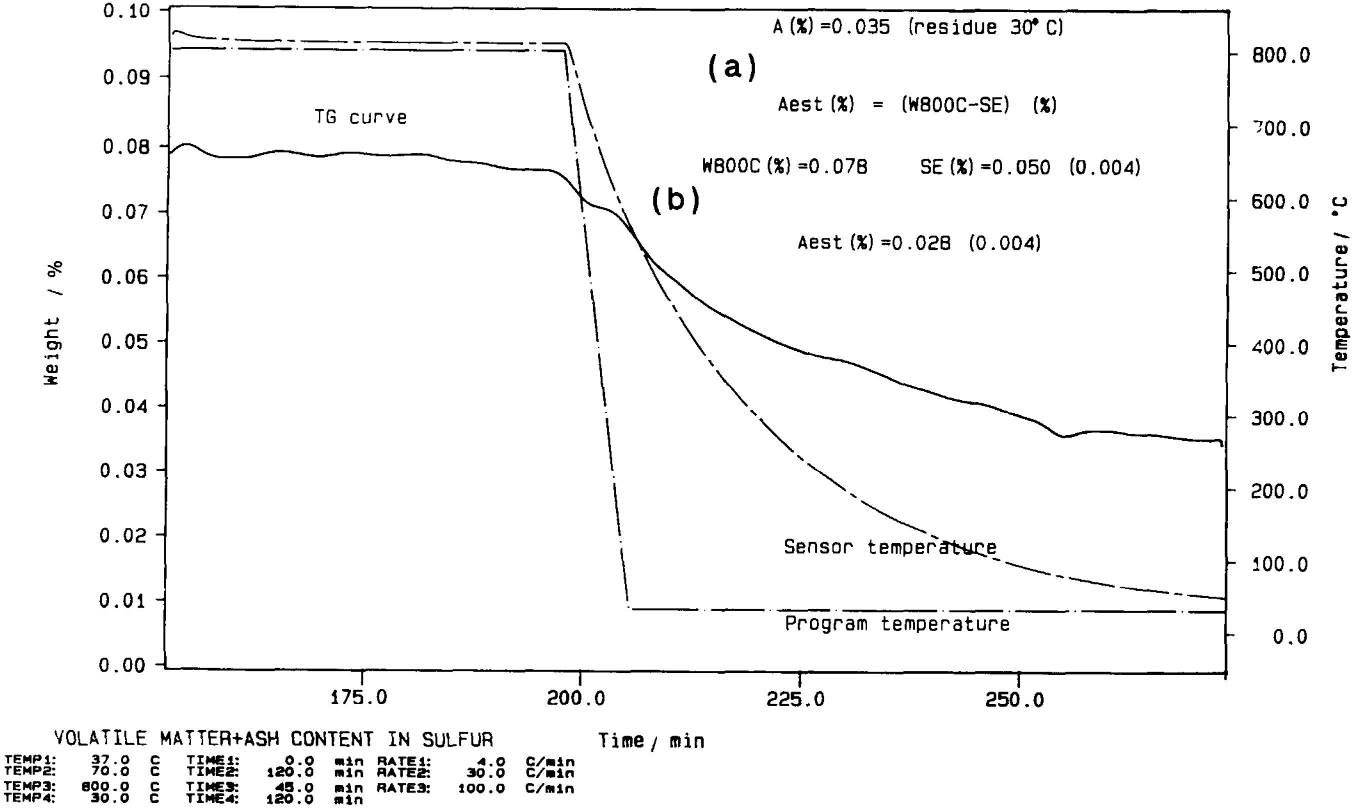

Fig. 3. Determination of the ash content in a sulfur sample using the TGIII method. a. Estimation of the ash content (A_{est}(%)) from the weight of the residue at 800°C, after ashing the sample for 45 min. b. Determination of the ash content from the weight of the residue at 30°C (A(%)). The TG curve together with the program and the sensor temperature curves are displayed.

Table 1
Percent volatile material of three sulfur samples, determined by different methods

Sample	Method	$V(\%)$			
		1	2	3	4
S1	VMD	0.032	0.054	0.032	0.040
	TGII	0.049	0.038	0.049	0.045
	TGIII	0.046	0.059	0.062	0.050
S2	VMD	0.037	0.063	0.048	0.038
	TGII	0.060	0.046	0.054	0.047
	TGIII	0.045	0.046	0.048	0.042
S3	VMD	0.048	0.067	0.046	0.037
	TGII	0.040	0.044	0.065	0.052
	TGIII	0.063	0.040	0.049	0.043

Table 2
Percent ash content of three sulfur samples, determined by different methods

Sample	Method	$A(\%)$			
		1	2	3	4
S1	ACD	0.048	0.020	0.047	0.040
	TGI	0.043	0.040	0.046	0.042
	TGII	0.037	0.028	0.027	0.039
	TGIII	0.042	0.030	0.038	0.044
S2	ACD	0.044	0.029	0.039	0.043
	TGI	0.045	0.043	0.026	0.025
	TGII	0.052	0.033	0.024	0.027
	TGIII	0.033	0.030	0.030	0.035
S3	ACD	0.039	0.029	0.038	0.039
	TGI	0.032	0.015	0.030	0.011
	TGII	0.032	0.026	0.036	0.022
	TGIII	0.033	0.033	0.031	0.023

Next, to unify the determinations of $V(\%)$ and $A(\%)$ in a single TG run and reduce the measuring time, the TGII test (see experimental section) was attempted. Again, the determinations were repeated four times on each sample (Tables 1 and 2) and the results for $V(\%)$ and $A(\%)$ were tested for significant differences compared with those yielded by the traditional methods (Tables 3 and 4).

Finally, the TGII method was modified by removing the intermediate isothermal stage to shorten even more the measuring time. This modification is called the TGIII method (Fig. 1). Once again, the determinations of $V(\%)$ and $A(\%)$ were repeated

Table 3
Determination of the volatile matter in S1, S2 and S3 (average values). Comparison of the results and the precision provided by the different thermogravimetric methods (TGII and TGIII) with those yielded by the standard test (ACD)

Sample	Method	$\langle V \rangle$(%)	s	df_F	F	s_p	df_t	t
S1	VMD	0.03950	0.01038	3				
	TGII	0.04525	0.00519	3	4.0	0.00820	6	0.991
	TGIII	0.05425	0.00750	3	1.9	0.00905	6	2.300
S2	VMD	0.04650	0.01207	3				
	TGII	0.05175	0.00655	3	3.4	0.00971	6	0.765
	TGIII	0.04500	0.00245	3	24.3	–	3.4(3)	0.240
S3	VMD	0.04950	0.01261	3				
	TGII	0.05025	0.01103	3	1.3	0.01184	6	0.089
	TGIII	0.04875	0.01021	3	1.5	0.01147	6	0.092

$F_{0.05/2,3,3} = 15.4$; $t_{0.05/2,6} = 2.45$; $t_{0.05/2,3} = 3.18$.

Table 4
Determination of the ash content in S1, S2 and S3 (average values). Comparison of the results and the precision provided by the different thermogravimetric methods (TGI, TGII and TGIII) with those yielded by the standard test (ACD)

Sample	Method	$\langle A \rangle$(%)	s	df_F	F	s_p	df_t	t
S1	ACD	0.04000	0.01340	3				
	TGI	0.04275	0.00250	3	28.7	–	3.3(3)	0.400
	TGII	0.03275	0.00613	3	4.8	0.01042	6	0.984
	TGIII	0.03850	0.00619	3	4.7	0.01044	6	2.450
S2	ACD	0.03875	0.00685	3				
	TGI	0.03475	0.01072	3	2.4	0.00899	6	0.630
	TGII	0.03400	0.01257	3	3.4	0.01012	6	0.664
	TGIII	0.03200	0.00245	3	7.8	0.00514	6	2.180
S3	ACD	0.03625	0.00486	3				
	TGI	0.02566	0.00929	3	3.7	0.00741	6	2.019
	TGII	0.02950	0.00551	3	1.3	0.00519	6	1.840
	TGIII	0.03000	0.00476	3	1.0	0.00481	6	1.838

$F_{0.05/2,3,3} = 15.4$; $t_{0.05/2,6} = 2.45$; $t_{0.05/2,3} = 3.18$.

four times on each sample (Tables 1 and 2, Figs. 2 and 3). The results obtained were compared with those obtained by the traditional methods and tested for significant differences (Tables 3 and 4).

Although at this point the measuring time had been considerably shortened, it could be reduced even more by eliminating the "cooling to 30°C step". This could be done if the ash content of the sample at 30°C (A(%)) could be estimated from the weight of the residue at 800°C ($W_{800^\circ C}(\%)$). To decide whether this cooling step was essential or not, $W_{800^\circ C}(\%)$ was related to $A(\%)$ for each determination on each sample using each TG test (Table 5). In every case, it was shown that the quantity $\Delta W(\%) = W_{800^\circ C}(\%) - A(\%)$ was constant (Tables 5 and 6). Then, a systematic difference in weight was observed. Once this systematic error is estimated ($\langle SE \rangle(\%)$), the ash content of the sample can be calculated from the measurement at 800°C ($A_{est}(\%)$) (Table 7, Fig. 3).

3.2. *Statistical analysis*

To determine whether the different thermogravimetric methods attempted for the determination of $V(\%)$ and $A(\%)$ yield the same results with the same precision as the methods currently used, a statistical analysis of the data was performed.

The calculated average values $\langle V \rangle(\%)$ and $\langle A \rangle(\%)$, and the corresponding variances s^2, obtained for each sample by the different TG methods, have been compared to those obtained by the standard methods. The comparison of the variances was performed through the F test and the comparison of the averages through the t test (both two-tailed and for a confidence level $\alpha = 0.05$). These tests are summarized in Tables 3 and 4, and establish whether there is any difference between the precision of the standard tests and the TG methods (their variances are equal for the chosen confidence level) and whether they yield the same results (the different methods provide average values for $\langle V \rangle(\%)$ and $\langle A \rangle(\%)$ that do not differ significantly for the chosen confidence level). From the data given in Tables 3 and 4, it can be concluded that the TG methods are at least as precise as the traditional tests. Moreover, it is also demonstrated that the results for $\langle V \rangle(\%)$ and $\langle A \rangle(\%)$ obtained by the TG experiments do not differ significantly from those yielded by the currently used methods.

Once it had been demonstrated that the TG methods provide the same information and with the same precision as the traditional tests, we tried to reduce even more the time required to obtain the results. The measuring time is notably increased by the final cooling step, which is necessary to determine the weight of the ashes at 30°C ($A(\%)$). For this reason, we attempted to eliminate this step by comparing the weight of the residue at 800°C ($W_{800^\circ C}(\%)$) to the weight of the ashes at 30°C ($A(\%)$). The difference between these quantities ($\Delta W(\%)$) was calculated for every experiment in order to determine if a constant value is obtained (Table 5). First, the statistical analysis was performed considering the different TG methods separately. For a given TG method, the variances corresponding to the values $\langle \Delta W(\%) \rangle$ of all the samples studied were confirmed to be homogeneous using the Cochran test [3] (confidence level $\alpha = 0.05$). Moreover, the Duncan separation of means [3] proved that the values $\langle \Delta W(\%) \rangle$ of all the samples do not differ significantly (significant range 5%, Table 6). Then, when using a given TG method, the calculated values $\langle \Delta W(\%) \rangle$ for all the samples are statistically equal and of the same precision, i.e., a constant difference in the weight of the residue is observed between 800 and 30°C. Finally, $\langle \Delta W(\%) \rangle$ and s^2 of each series of measurements corresponding to all the samples and all the TG methods were compared to establish whether or not a constant difference in the weight of the ashes at 800 and 30°C is observed for the whole of the TG data. The Cochran test proved that all the variances were homogeneous ($\alpha = 0.05$, Table 5) and the Duncan separation of means confirmed

Table 5

Calculated differences $\Delta W(\%)$ between the weight of the residue at 800 and 30°C. The average value of this parameter together with the standard deviation (in parentheses) for each series of determinations on each sample, applying the different thermogravimetric methods, are included. In the lower part, the comparison of the variances through the Cochran test is shown

Sample	TGI				TGII				TGIII			
	$W_{800^\circ C}(\%)$	$A(\%)$	$\Delta W(\%)$	$\langle \Delta W \rangle(\%)$ (s)	$W_{800^\circ C}(\%)$	$A(\%)$	$\Delta W(\%)$	$\langle \Delta W \rangle(\%)$ (s)	$W_{800^\circ C}(\%)$	$A(\%)$	$\Delta W(\%)$	$\langle \Delta W \rangle(\%)$ (s)
S1	0.085	0.043	0.042		0.085	0.037	0.048		0.092	0.042	0.050	
	0.078	0.040	0.038	0.04075	0.078	0.028	0.050	0.04875	0.086	0.030	0.056	0.05100
	0.086	0.046	0.040	(0.0022173)	0.073	0.027	0.046	(0.0022173)	0.085	0.038	0.047	(0.0045825)
	0.085	0.042	0.043		0.090	0.039	0.051		0.088	0.044	0.044	
S2	0.090	0.045	0.045		0.100	0.052	0.048		0.082	0.033	0.049	
	0.075	0.026	0.049	0.04150	0.080	0.033	0.047	0.04675	0.083	0.030	0.053	0.04925
	0.075	0.043	0.032	(0.0073257)	0.068	0.024	0.044	(0.0018929)	0.082	0.030	0.052	(0.0045000)
	0.065	0.025	0.040		0.075	0.027	0.048		0.078	0.035	0.043	
S3	0.083	0.032	0.051		0.077	0.032	0.045		0.083	0.033	0.050	
	0.070	0.015	0.055	0.0493	0.068	0.026	0.042	0.04475	0.082	0.033	0.049	0.05075
	0.072	0.030	0.042	(0.0066583)	0.081	0.036	0.045	(0.0020615)	0.080	0.031	0.049	(0.0028722)
	0.063	0.011	0.052		0.069	0.022	0.047		0.078	0.023	0.055	
					Cochran test							
Individual methods	$g = 0.5215$, $g_{0.05,4,3} = 0.7977$ Homogeneity of variances $s_p = 0.005857$				$g = 0.3871$, $g_{0.05,4,3} = 0.7977$ Homogeneity of variances $s_p = 0.002061$				$g = 0.4242$, $g_{0.05,4,3} = 0.7977$ Homogeneity of variances $s_p = 0.004062$			
Joined methods					$g = 0.3249$, $g_{0.05,4,9} = 0.4027$ Homogeneity of variances $s_p = 0.004284$							

Table 6
Duncan's separation of means procedure applied to the average values of the differences between the weight of the residue at 800°C and the weight of the residue at 30°C $\langle \Delta W(\%) \rangle$

	Individual methods								
	TGI			TGII			TGIII		
s_p		0.005857			0.002061			0.004062	
df		9			9			9	
for samples of		2	3		2	3		2	3
significant ranges		3.20	3.34		3.20	3.34		3.20	3.34
least significant ranges		0.01874	0.01956		0.00659	0.00688		0.01299	0.01357
Averages	*A*	*B*	*C*	*A*	*B*	*C*	*A*	*B*	*C*
	0.0493	0.0415	0.04075	0.04875	0.04675	0.04475	0.05100	0.05075	0.04925
$A-C$	0.00855 is not significant			0.00400 is not significant			0.00175 is not significant		
$A-B$	0.00780 is not significant			0.00200 is not significant			0.00025 is not significant		
	Joined methods								
s_p					0.004284				
df					27				
for samples of		2	3	4	5	6	7	8	9
significant ranges		2.905	3.050	3.135	3.205	3.265	3.300	3.335	3.355
least significant ranges		0.01244	0.01306	0.01343	0.01373	0.01399	0.01414	0.01429	0.01437
Averages	*A*	*B*	*C*	*D*	*E*	*F*	*G*	*H*	*I*
	0.0510		0.05075	0.0	0.04925	0.04875	0.0	0.04475	0.041500
$A-I$					0.01025 is not significant				
$A-H$					0.00950 is not significant				
$A-G$					0.00625 is not significant				
$\ldots A-B \ldots$					0.00025 is not significant				

Table 7
Comparison of the results obtained for the estimation of the ash content from the weight of the residue at 800°C, using the TGIII test, and the results yielded by the traditional ACD methods

Sample	$\langle W_{800\,\mathrm{C}}\rangle$(%)	s	$\langle A_{\mathrm{est}}\rangle$(%)	s_{est} [a]	df_F	F	s_{p}	df_t	t [b]
S1	0.08775	0.0030956	0.03800	0.0070297	3	3.6	0.010699	6	0.264
S2	0.08125	0.0022173	0.03150	0.0061514	3	1.2	0.006510	6	1.575
S3	0.08075	0.0022173	0.03100	0.0061514	3	1.6	0.005543	6	1.339

$\langle SE\rangle$(%) = 0.04975 (systematic error: average value of all the differences ΔW for all the determinations using the *TGIII* test). $s_{\mathrm{SE}} = 0.0039341$ (standard deviation of the calculated systematic error). $\langle A_{\mathrm{est}}\rangle(\%) = [\langle W_{800\,\mathrm{C}}\rangle - \langle \mathrm{SE}\rangle](\%)$.

$s_{\mathrm{est}} = s + s_{\mathrm{SE}}$. $F_{0.05/2,3,3} = 15.4$. $t_{0.05/2,6} = 2.45$.

[a] $(s_{\mathrm{est}})^2$ is compared to s^2 corresponding to the ACD test, for each sample (Table 4).

[b] $\langle A_{\mathrm{est}}\rangle$(%) is compared to $\langle A\rangle$(%) obtained by the ACD test, for each sample (Table 4).

that the average values $\langle \Delta W(\%) \rangle$ do not differ significantly for the different methods (significant range 5%; Table 6). Then, it is concluded that the difference between the weight of the residue at 800° and 30°C can be considered as constant, i.e. there is a systematic error whose value and deviation can be estimated.

Because the TGIII method is the least time-consuming, it will be adopted for the determination of $\langle V(\%) \rangle$ and $\langle A(\%) \rangle$ in polymeric sulfur specimens. Then, the systematic error will be calculated taking *only* into account the results obtained using this method. The error introduced when weighing the residue at 800°C has been calculated as the average value of all the differences of weight $\Delta W(\%) = W_{800^\circ C}(\%) - A(\%)$ for this method, being $\langle SE \rangle(\%) = 0.04975$ ($s = 0.0039341$). To estimate the weight of the residue at room temperature for each sample ($A_{est}(\%)$), $\langle SE \rangle(\%)$ should be subtracted from $W_{800^\circ C}(\%)$ (Fig. 3). If the results of $A_{est}(\%)$ were equal to those obtained by the traditional method, the estimated values could be used as a good measurement of the ash content, and thus the measuring time required to determine this parameter could be noticeably reduced.

In order to confirm whether or not the results obtained for the ash content of a sulfur sample when using the ACD method ($\langle A \rangle(\%)$, Table 4) are equal to those estimated from the measurement at 800°C using the TGIII method ($\langle A_{est} \rangle(\%)$, Table 7), the corresponding variances and average values must be compared (Table 7). For each sample, the variances compared through the F test are equal; in addition, the average values $\langle A \rangle(\%)$ and $\langle A_{est} \rangle(\%)$ compared through the t test do not differ significantly for a confidence level $\alpha = 0.05$. Thus, it can be concluded that the ash content of a polymeric sulfur specimen can be estimated from the weight of the residue at 800°C, the result and precision of this determination being equal to those provided by the traditional test.

4. Conclusions

A new thermogravimetric method (TGIII) for the determination of the volatile matter and ash content of a sulfur specimen has been developed. This method allows the determination of both parameters in a single operation, providing at least the same information and with the same precision as that obtained with the currently used methods. It is worth noting that this TG test is faster (measuring time 3 h) and that the amount of sample required is considerably smaller (120 mg) than in the traditional tests. However, as the sample is weighed only at the beginning of the experiment, the volatile matter and the ash content being determined directly on the TG curve, the operation time is considerably reduced.

Acknowledgments

The authors wish to thank Dr. F. Temprano and his group for supplying the samples. We also are indebted to Mrs. González-García for her collaboration in this work.

References

[1] Standard Test Method for Rubber Chemicals. Determination of volatile material. ASTM, Designation: D 4571-89.

[2] Standard Test Method for Rubber Chemicals. Determination of ash content. ASTM, Designation: D 4574-89.

[3] R.L. Anderson, Practical Statistics for Analytical Chemists, Van Nostrand Reinhold Co., New York 1987.