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## **Inconsistent chemical data on phlogopite: analyses and genesis of phlogopites from Madagascar**

by *W. B. Stern*<sup>1</sup> and *H. H. Klein*<sup>2</sup>

### **Abstract**

Precambrian pyroxenites in South Madagascar contain phlogopites being today worldwide a main source of phlogopite used in the electrical industry. Literature data on the chemistry of phlogopites from the two mines of Ampandrandrave and Sakamasy are scarce, and highly inconsistent. At both mining sites well-located samples were collected and analyzed by means of energy dispersive X-ray fluorescence spectrometry. Significant, but small chemical differences between phlogopites from the two sites exist. The local chemical variation turns out to be small, often smaller than the analytical variation (error). Thus, no interdependence between chemical composition and sample localization was detectable.

Based upon new phlogopite analyses and literature data of pyroxenites and diopside from the two sites, a mass balance has been calculated assuming a metasomatic transformation of tectonically affected pyroxenites (metasomatic supply of 4 to 4.5% K<sub>2</sub>O, 0 to 1.5% MgO, ± Al<sub>2</sub>O<sub>3</sub>) as educt, and phlogopites (60 to 70% phlogopite, 5 to 10% fassaitic diopside, 20 to 30% calcite, and 0 to 5% anhydrite) + eluate (Si, Ca) as product, and keeping the volume of educt and product constant. Fluorine present in phlogopites between 1.5 and 3%, but not in pyroxenites, is supposed to be an essential agent in this metasomatic process, which was probably connected with the intrusion of discordant pegmatites present at Ampandrandrave and Sakamasy as well.

*Keywords:* phlogopite, chemical analysis, metasomatism, mineral deposits, Malagasy Republic

### **Introduction**

Madagascar is the main world source of all forms of phlogopite mica, and its output of sheet mica (splittings) amounts to 5% (1972), 10% (1968) respectively of the world splitting production of muscovite/phlogopite (PETKOV, 1972). The major part of the actually available phlogopite mica originates from mines located in the south of Madagascar, today particularly from Ampandrandrave and Sakamasy (fig. 1). Below-ground mining operations in Precambrian pyroxenites began in 1921, and 1935 respectively.

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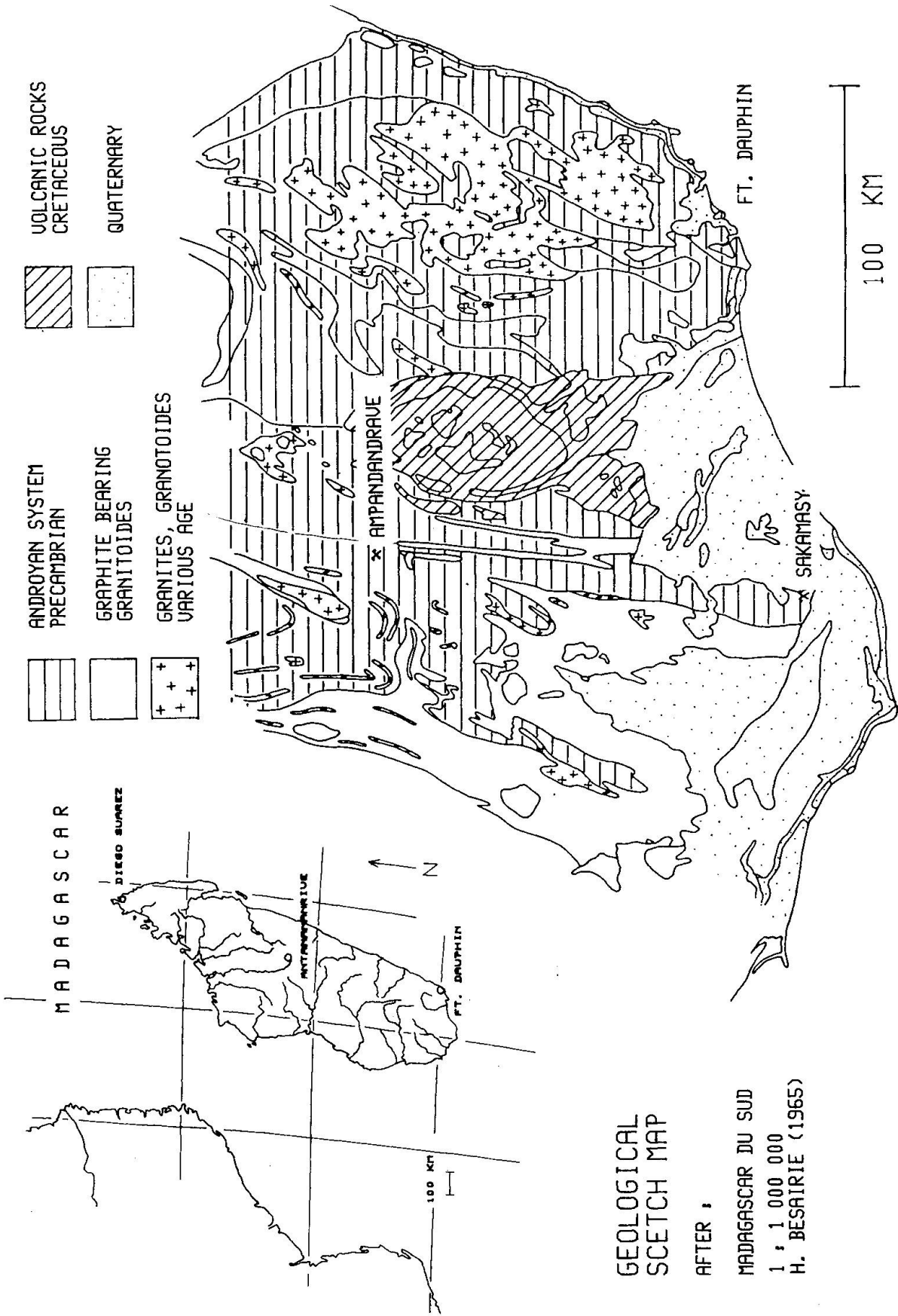


Fig.1 Geological sketch map of southern Madagascar.

The whole production is consumed by the electro industry abroad, representing therefore an important factor for the Malagasy economy.

Published chemical analyses of phlogopite from Ampandandrave (N = 6) and Sakamasy (N = 1) are scarce (JAKOB & PARGA, 1932; DE LA ROCHE, 1963; JOO, 1972) and are remarkably inconsistent: whereas the analyses of JAKOB et al. and DE LA ROCHE (op. cit.) represent true phlogopite micas, part of the more recent data of JOO cannot be interpreted as end members of the trioctahedral

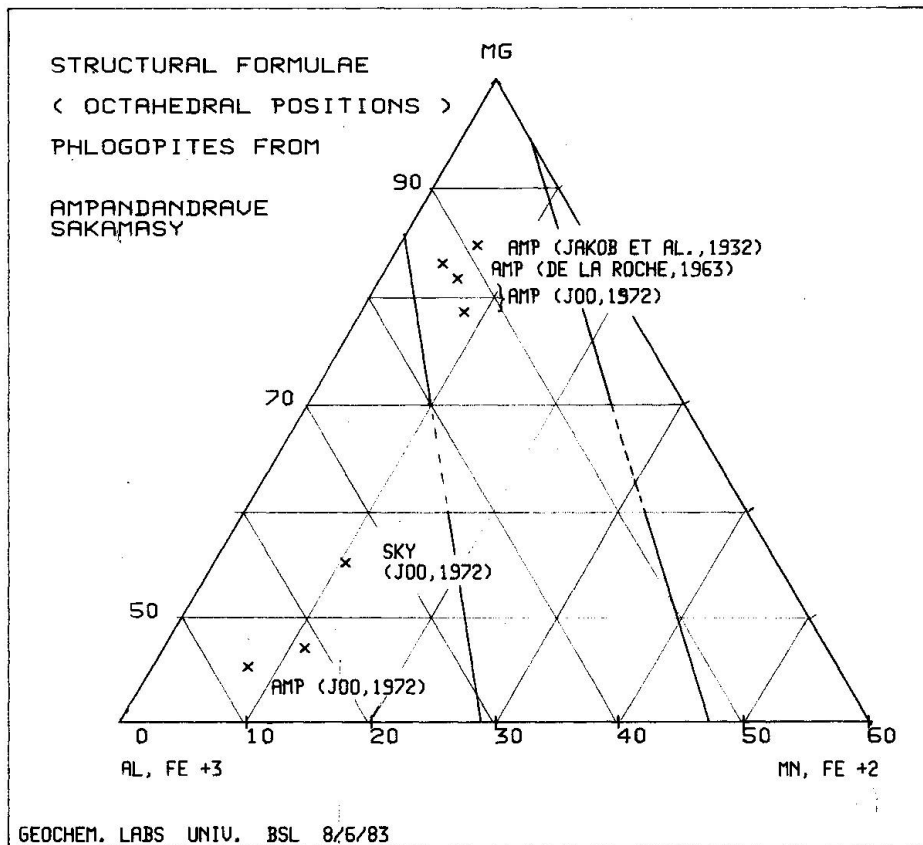


Fig. 2 Phlogopites from Ampandandrave and Sakamasy, data taken from literature.

Mg-Fe mica series (fig. 2). Are there striking distinctions due to an eventual inhomogeneity of the phlogopite deposits (in connection with the controversial genesis of these deposits, DE LA ROCHE, 1963) or due to other reasons?

According to the genetical history - metamorphous/metasomatic and/or magmatic - chemical gradients within one and the same deposit are conceivable; detailed indications of the sampling procedure are missing, however, in the cited literature. In general the chemical variation of phlogopite in its solid solution series within one and the same deposit is unknown (the same holds for muscovite, too).

### Geological outline

The productive phlogopite deposits of southern Madagascar are located in steeply dipping pyroxenite lamellae (width some few meters, length up to some kilometers) together with alternating beds of gneisses and leptynites of the Precambrian Androyan System affected by an archaic metamorphism in granulite facies (radiometric age 2100–2500 m.a., DELBOS, 1965).

Based above all on geochemical arguments the pyroxenites are interpreted since the works of LACROIX, 1941, DE LA ROCHE and JOO (op. cit.) as metasediments of formerly lagoonous marns.

The rock series is affected by a system of fissures – subparallel and perpendicular to the strike. The enriched occurrence of phlogopite in form of pockets and lenses is related to the tectonic pattern of the host rocks, but the phlogopite itself is tectonically not affected. Pegmatites strike discordantly through the rock series. Radiometric age determinations of the phlogopite (K/Ar, Rb/Sr) revealed 530 m.a. which corresponds to the last pan-african plutonic cycle (LEUTWEIN, 1964).

### Sampling

To clarify the question of the chemical variance of phlogopite from one and the same deposit, samples were collected by the authors in Ampandandrave and Sakamasy, and later analyzed. The test material covers all accessible tunnels in horizontal/lateral and vertical direction (fig. 3). 17 fresh, unaltered phlogopites from each mine were sampled. Help and assistance is gratefully acknowledged to M Fauret/Ampandandrave and M Renot/Sakamasy.

### Analysis

The analyses were carried out by means of energy-dispersive X-ray fluorescence spectrometry (EDS-XFA). Table I indicates the analytical parameters, STERN et al. 1982. The calculated variation ( $3\sigma$ , 99% confidence level) corresponds at Sakamasy to the analytical error (based upon counting statistics) for all main constituents (Si, Al, Fe, Mg, K and F) with exception of water, see table II. Thus, no chemical gradation or zonation whatsoever can be ascertained at Sakamasy. At Ampandandrave Fe and Mg vary within the range of the respective analytical errors, whereas the variation of the other elements is slightly outside the range of analytical error. The chemical variation, however, cannot be correlated with the locality of sampling regarding lateral or vertical directions – the variation seems to be random.

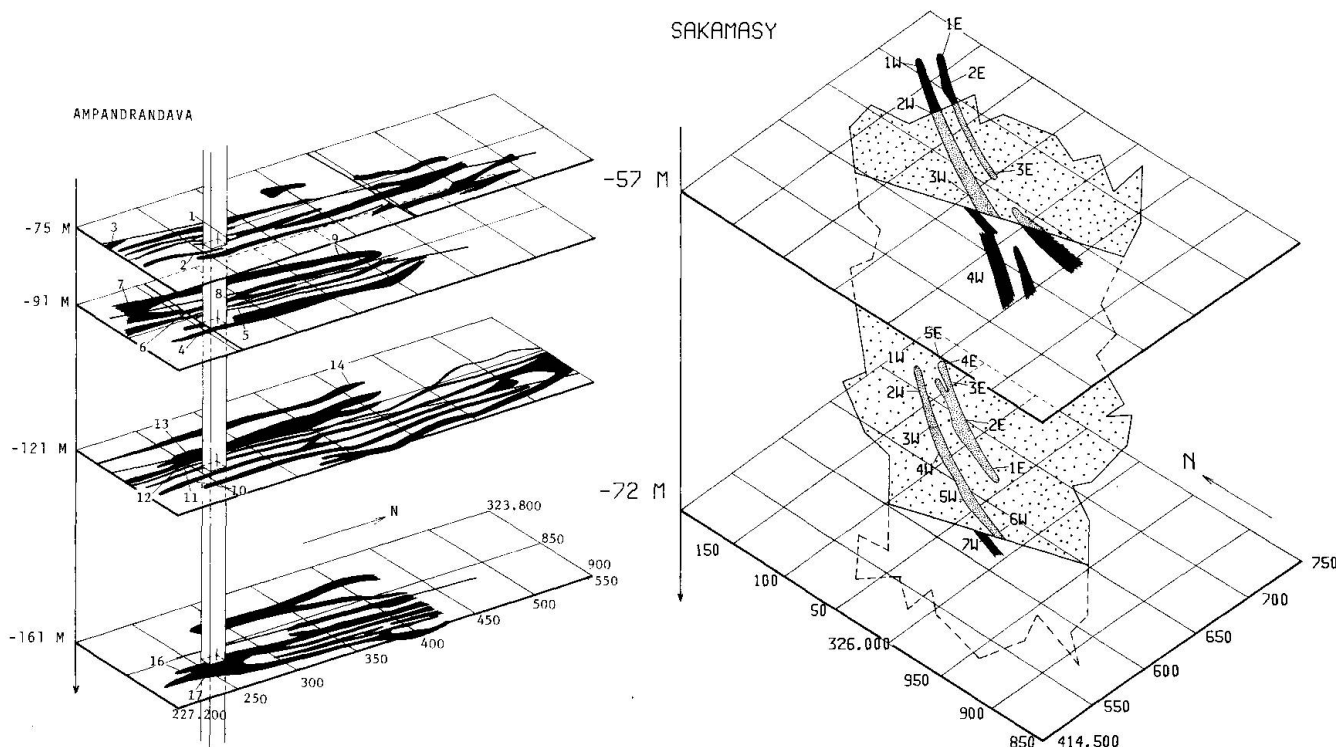


Fig. 3 The mining sites of Ampandrandava and Sakamasy (schematic) with indications on sampling.

The large range of chemical variation as reported by JOO (1972) for phlogopites originating from Sakamasy and Ampandrandava is not confirmed by these analytical series. Most probably the published data represent incorrect analyses and therefore are the consecutive crystal-chemical deductions (JOO, 1972, p. 12 ff.) meaningless.

Whereas the analysis of main constituents leads to results displaying quantifiable errors, the determination of volatile components remains fairly tricky.

Table I Analytical Parameters.

Apparatus	Energy dispersive X-ray fluorescence spectrometer TN-1710 of Tracor Northern. Detector 150 eV resolution. 2000 channel analyzer. Spectrometer designed at Geochem. Labs / Basle University ( Min.-petr. Institute )
Excitation	Ag tube, 21 kV, 0.05 mA. 200 sec counting time (life). Powdered and pressed samples ( STERN, 1979 ).
Analysis	software "Super-ML, matrix correction RH" by Tracor Northern, and "energy calibration, data reduction, plotting" by Gloor Instruments. Reference file 5 / RH 5 on fl 80. Silicate rocks and minerals as standards. Fluorine: ion selective electrodes ( CAMMANN, 1977 ).

Table II Average composition of 17 phlogopites from Ampandandrave and Sakamasy (EDS - XFA) wt-%.

Ampandandrave	$\bar{x}$ N=17	s	CoV*	CoV**	JAKOB et al. 1932
SiO <sub>2</sub>	38.28	0.426	3.3	1.0	38.4
Al <sub>2</sub> O <sub>3</sub>	16.01	.479	9.0	2.8	16.7
Fe <sub>2</sub> O <sub>3</sub>	6.37	.175	8.2	5.8	4.5
MnO	0.18	.015	25.	13.4	0.15
MgO	23.41	.340	4.4	4.5	23.4
K <sub>2</sub> O	9.97	.123	3.7	1.2	10.9
TiO <sub>2</sub>	1.15	.090	24.	2.7	1.69
H <sub>2</sub> O	3.20	.996	92.	11.8	3.2
F	1.53	.201	39.	18.7	.7
Sakamasy					
SiO <sub>2</sub>	38.11	0.026	0.2	1.0	
Al <sub>2</sub> O <sub>3</sub>	16.30	.400	2.5	2.8	
Fe <sub>2</sub> O <sub>3</sub>	6.97	.263	11.3	5.8	
MnO	0.07	.011	43.	13.4	
MgO	23.29	.369	4.8	4.5	
K <sub>2</sub> O	10.21	.062	1.8	1.2	
TiO <sub>2</sub>	.90	.068	23.	2.7	
H <sub>2</sub> O	2.55	.556	66.	11.8	
F	2.41	.183	23.	18.7	
* Coefficient of variation = $3\sigma \cdot 100 / \bar{x}$ evaluation based upon 17 single determinations					
** Coefficient of variation = $3\sigma \cdot 100 / \bar{N}$ based upon counting statistics, connected with analytical error					

The determined loss on ignition is not only dependent on the calcination temperature but also on the calcination time as well as on the grain size distribution of a sample (fig. 4). Since only calcination time and temperature are usually kept constant (they differ, however, from one laboratory to another), and since little is known about the actual grain size distribution, published data on volatile contents are inconsistent and subject to remarkable errors, see table III.

Phlogopites of Madagascar contain fluorine as a main component which is nowadays accurately determined by means of ionselective electrodes, whereas

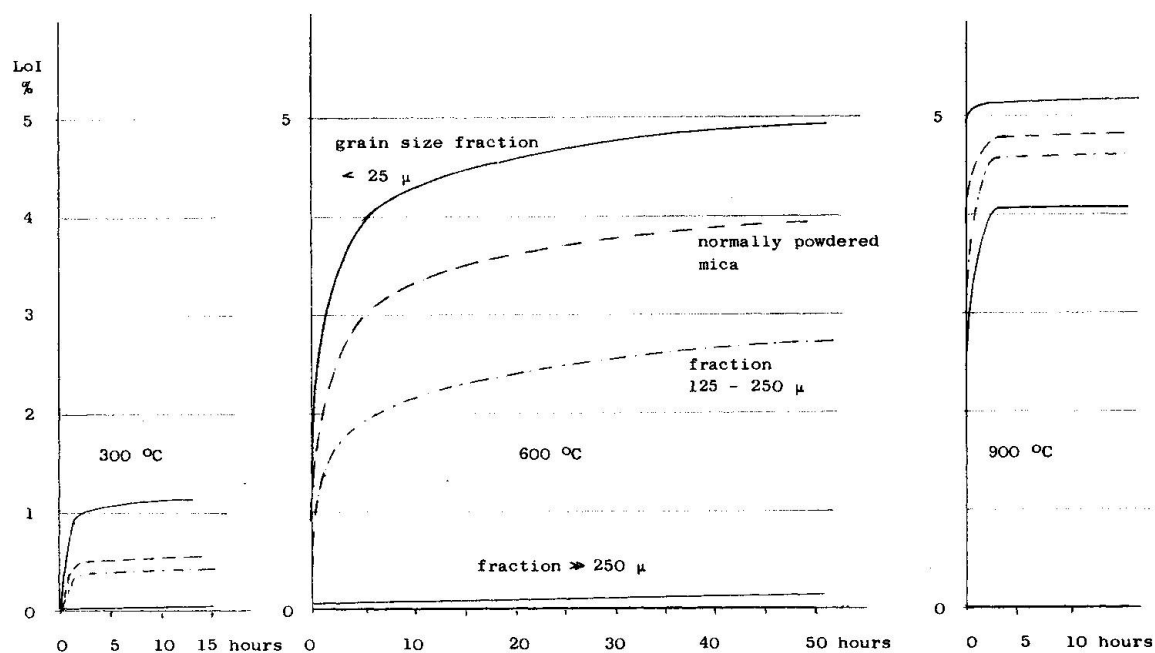


Fig. 4 Weight loss of mica during calcination at different temperatures, static thermogravimetry. For small grain size fractions a creeping weight loss is observed (after KLEIN et al., 1982).

the old distillation methods gave often erroneous results (VGENOPOULOS, 1971). Whether fluorine is partially or entirely removed from phlogopite during calcination depends on the ignition routine used, fig. 5.

Differences in  $H_2O$  and volatile contents found by different laboratories analyzing the same, homogeneous sample, are due to the different calcination methods, whereas the variation of actual fluorine determinations may represent real chemical variations. Phlogopites from Sakamasy are significantly richer in

Table III Analytical variation of volatiles in silicate standards.

standard	NIM-G	NIM-S	NIM-L	NIM-N	NIM-P	NIM-S	
mean	0.77	0.34	2.77	0.64	0.19	0.20	Loss on ignition
relative error, %	11.5	34.1	3.1	186	226	383	
mean	0.58	0.29	2.44	0.41	0.35	0.38	water, $H_2O^+$
relative error, %	42.8	46.4	8.4	54	75	48	
mean	0.14	0.15	0.24	0.11	0.05	0.10	moisture, $H_2O^-$
relative error, %	77	86	62	94	99	79	

relative error = coefficient of variation =  $1\sigma \cdot 100 / \text{mean}$

Data basis : Report No. 1351 of the National Institute for Metallurgy,  
Johannesburg 1972



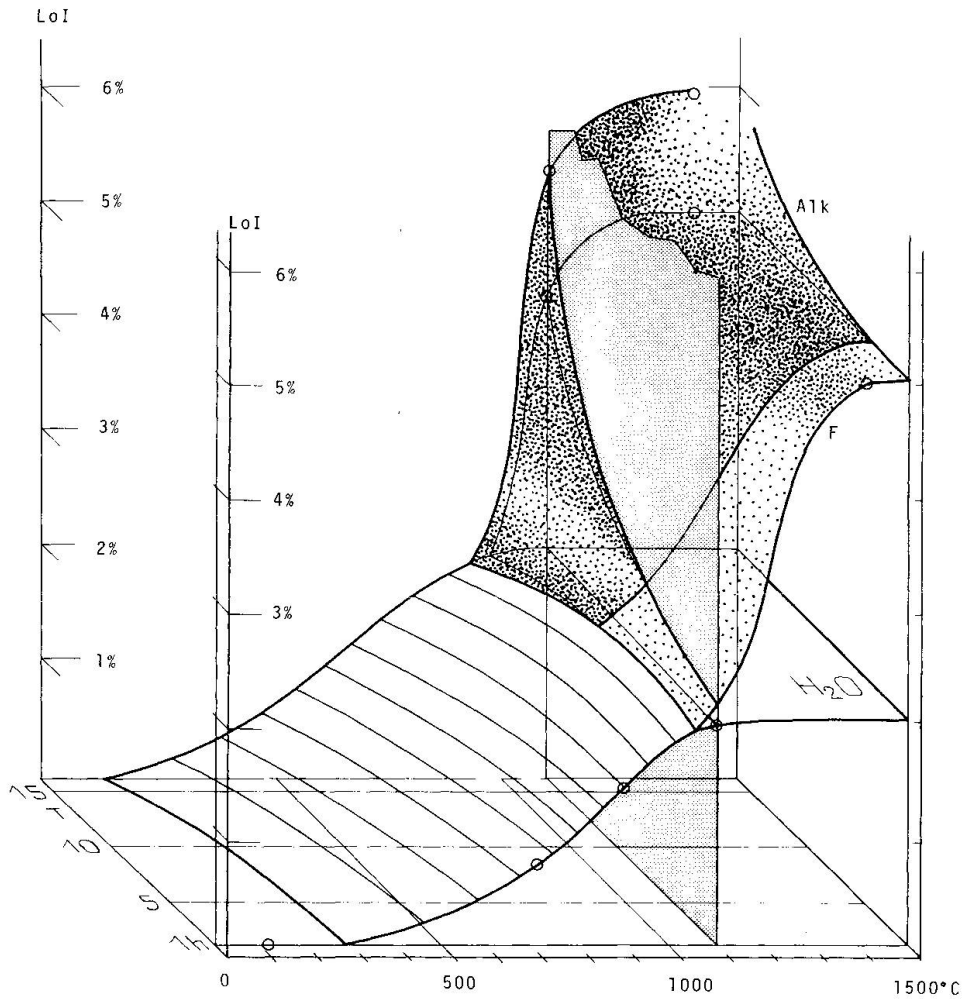


Fig. 5 Phenomena during calcination of phlogopite (SKY 1/581). Mica chips ground under norm conditions, average grain size less than  $60\ \mu\text{m}$ . Experimentally determined points are symbolized by circles, schematic. Details of the calcination routine, like ignition time and temperature influence severely the loss on ignition (LoI) found. Above  $1100^\circ\text{C}$  and 1 hour calcination time fluorine and alcalies begin to disappear.

fluorine than those of Ampandandrave, variations, however, being considerable at both mines. A dependancy of the fluorine content in function of the sample location (depth, host rock etc.) could not be established.

### Results

Seventeen phlogopite analyses per mine from both, Ampandandrave and Sakamasy, display significant differences between the two deposits, but only little variations within a site being—in case of the main constituents—in the range of the analytical errors, table IVa/b. This proven homogeneity (fig. 6) of the two phlogopite deposits asks for a new genetical interpretation.

Phlogopites from Madagascar

Table IVa Chemical analyses (EDS - XFA) of phlogopites from Ampandandrave.

A M P A N D R A N D A V E		75-1	75-2	75-3	91-4	91-5	91-6	91-7	91-8	91-9	121-10	121-11	121-12	121-13	121-14	150-15	161-16	161-17
IX		5582	5583	5584	5585	5586	5587	5588	5589	5590	5591	5592	5593	5594	5595	5596	5597	6017
SiO <sub>2</sub>		38.6	38.9	39.0	38.3	38.6	38.2	37.9	37.8	37.9	38.3	38.7	38.3	37.7	38.8	38.3	37.6	37.7
Al <sub>2</sub> O <sub>3</sub>		16.4	16.0	16.4	16.1	16.0	16.3	15.8	15.5	16.5	16.2	14.7	15.6	15.9	16.7	16.4	16.0	15.6
Fe <sub>2</sub> O <sub>3</sub>		2.71	2.52	2.51	2.47	2.44	2.37	2.21	2.47	2.06	2.52	2.50	2.15	2.16	2.39	2.32	2.15	2.36
FeO		3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6	3.6
MnO		.16	.20	.16	.17	.18	.18	.16	.18	.21	.17	.16	.17	.19	.19	.18	.17	.17
MgO		23.6	23.7	23.6	23.3	23.1	23.6	23.2	23.0	23.5	23.4	24.1	23.9	23.2	23.3	23.6	22.9	22.9
CaO		.02	.0	.13	.06	.0	.05	.04	.0	.13	.12	.0	.03	.0	.0	.05	.0	.01
Na <sub>2</sub> O		.4	.4	.3	.3	.2	.3	.4	.4	.2	.3	.3	.3	.3	.2	.2	.3	.3
K <sub>2</sub> O		10.06	9.98	10.09	10.01	10.15	9.98	9.90	9.83	9.89	9.86	9.76	9.89	9.92	10.07	10.12	9.96	9.77
TiO <sub>2</sub>		1.26	1.33	1.21	1.22	1.13	1.18	1.23	1.15	.97	1.21	1.06	1.06	1.12	1.07	1.17	1.15	1.05
Rb <sub>2</sub> O		.07	.06	.06	.06	.06	.07	.07	.07	.04	.07	.07	.07	.06	.08	.07	.06	.05
H <sub>2</sub> O		1.5	1.2	1.3	3.9	3.3	3.2	4.2	4.5	3.6	3.9	3.5	3.8	3.7	2.9	4.0	3.3	3.6
Cl		.08	.06	.06	.04	.06	.05	.04	.05	.07	.08	.08	.04	.05	.06	.06	.03	.05
F		1.6	1.7	1.55	1.2	.95	1.7	1.7	1.55	1.65	1.4	1.45	1.65	1.7	1.55	1.45	1.6	1.65
O = F		.66	.70	.64	.49	.39	.70	.70	.64	.68	.57	.59	.68	.70	.64	.59	.66	.68
Sum		99.32	98.95	99.33	100.74	99.38	100.08	99.75	99.46	99.64	100.19	99.32	99.88	98.90	100.27	100.93	98.16	98.13

Table IVb Chemical analyses (EDS - XFA) of phlogopites from Sakamasy.

	57-1E	57-1W	57-2E	57-2W	57-3E	57-3W	57-4W	72-1E	72-2E	72-2W	72-3E	72-3W	72-4E	72-4W	72-5W	72-6W	72-7W
IX	6000	5599	6004	6005	6008	6009	6012	5598	6002	6003	6007	6006	6011	6010	6014	6015	6016
SiO <sub>2</sub>	38.3	38.4	38.2	38.1	38.4	38.0	37.9	38.4	38.7	38.1	38.4	38.0	38.0	37.9	37.7	37.8	37.4
Al <sub>2</sub> O <sub>3</sub>	16.4	16.7	16.9	16.3	16.9	16.4	16.3	16.3	15.7	16.9	15.8	16.0	15.9	16.0	16.6	15.8	16.2
Fe <sub>2</sub> O <sub>3</sub>	2.14	1.78	2.47	2.27	2.24	2.16	2.63	2.56	2.18	2.19	2.74	2.21	2.74	2.21	2.20	2.02	2.56
FeO	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2	4.2
MnO	.06	.09	.08	.08	.08	.08	.06	.07	.08	.09	.07	.05	.07	.08	.07	.08	.07
MgO	23.4	23.8	23.9	23.2	23.7	23.5	22.8	23.5	23.7	23.2	23.4	23.0	22.9	23.2	22.8	23.2	22.7
CaO	.0	.02	.16	.0	.0	.0	.07	.11	.03	.24	.0	.0	.12	.0	.0	.01	.02
Nb <sub>2</sub> O	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1	.1
K <sub>2</sub> O	10.20	10.27	10.29	10.22	10.25	10.25	10.14	10.31	10.23	10.20	10.22	10.19	10.09	10.19	10.11	10.25	10.14
TiO <sub>2</sub>	.85	.87	.75	.91	.86	.90	.84	.96	1.00	.82	.97	.95	.96	.94	.85	.98	.94
Rb <sub>2</sub> O	.05	.07	.07	.07	.06	.07	.06	.07	.06	.05	.08	.07	.08	.08	.05	.08	.09
H <sub>2</sub> O	2.3	3.1	2.0	2.8	2.1	1.9	2.5	3.9	1.9	2.6	2.2	2.6	2.4	2.4	3.2	2.0	2.0
Cl	.07	.08	.08	.08	.08	.07	.09	.07	.08	.07	.07	.07	.08	.06	.08	.07	.07
F	1.8	2.5	2.5	2.5	2.5	2.6	2.3	2.3	2.6	2.4	2.5	2.3	2.4	2.4	2.4	2.5	2.5
O = F	.74	1.03	1.03	1.03	1.03	1.07	.94	.94	1.07	.98	1.03	.94	.98	.98	.98	1.03	1.03
Sum	99.03	100.85	101.60	99.70	100.34	99.06	98.95	101.91	100.13	100.26	99.08	98.71	99.06	98.78	99.38	98.06	97.96

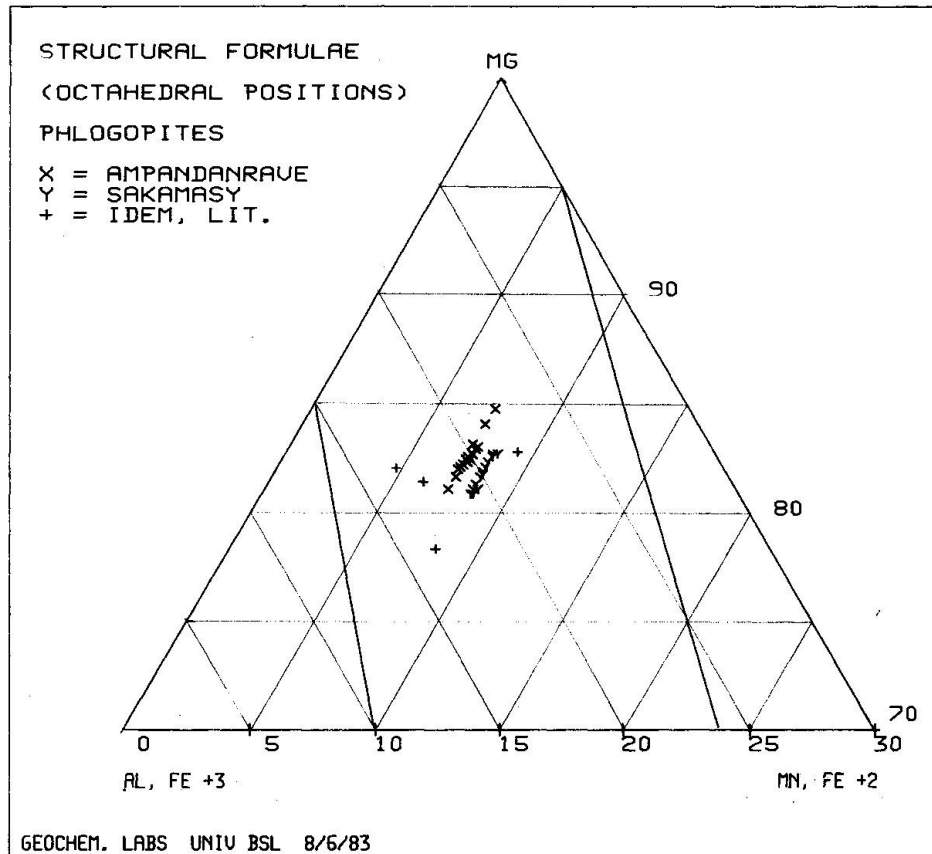


Fig. 6 Phlogopites from Ampandandraue and Sakamasy, new data, and data taken from literature (cross after JAKOB et al. (1932), DE LA ROCHE (1963) and JOO (1972)).

DE LA ROCHE (1963) assumes a metasomatic transformation of the pyroxenite by supply of potassium and magnesium in a fluid phase. His different attempts to balance the reaction are only partially satisfying since these calculations were based upon Al-free diopside. The actual pyroxenites consist up to 90% of fassaite containing 7 to 11%  $\text{Al}_2\text{O}_3$  (MAJMUNDAR, 1971), which affects the balance heavily.

JOO (1972) postulates a single phase genesis: transformation of lagoonous marns into pyroxenite by metamorphism in granulite facies and at the same time partial/differential transformation (hydrotogenesis) of the pyroxenite by supply of marine-inherited potassium, and a fluid phase.

New data suggest a somewhat modified genesis, which is still based on the idea of LACROIX (1941):

- Precambrian pyroxenites - chemically and mineralogically identical with the actual ones (granulite facies metamorphism between 2100 and 2500 m.a.) - are effected by tectonic events: fissures subparallel and perpendicular to the strike of the host rocks are formed, inducing an increased mobility in the tectonically affected zones.



Table VI Calculated derivation of phlogopitite from pyroxenite, Sakamasy Mine (wt-%).

	educt		product		calculated phlogopitite					balance (pyroxenite-sum)
	pyroxenite N=37 +1σ	diopside N=3 +1σ	phlogopite N=17 +1σ	8 di	54 phl	26 cc	sum			
SiO <sub>2</sub>	45.3 +1.6	43.7 +3.4	38.1 +0.3	3.5	20.6	24.1	+ 21.2			
Al <sub>2</sub> O <sub>3</sub>	10.1 4.4	16.7 3.8	16.3 .4	1.3	8.8	10.1	.0			
Fe <sub>2</sub> O <sub>3</sub>	4.3 2.6	4.7 .5	6.9 .3	.4	3.7	4.1	+ .2			
MgO	11.3 2.5	8.1 4.1	23.3 .4	.6	12.6	13.2	- 1.9			
CaO	24.4 1.1	23.1 3.1	.0	.2	.0	14.6	+ 9.6			
Na <sub>2</sub> O	.6 .4	.2 .2	.3 .1	.0	.2	.2	+ .4			
K <sub>2</sub> O	.8 .6	.8 .8	10.2 .1	.0	5.4	5.4	- 4.6			
TiO <sub>2</sub>	.5 .2	.4 .3	.9 .1	.0	.5	.5	.0			
H <sub>2</sub> O	2.4 1.1	2.0 .9	2.6 .6	.2	1.4	1.6	+ 0.8			
F			2.4 .2		1.3	1.3	- 1.3			
CO <sub>2</sub>						11.4	- 11.4			
educt			product				+ 32.2 "eluate"			
101 pyroxenite (84%)			88 phlogopitite (73%)				- 19.2			
19.2 K <sub>2</sub> O,CO <sub>2</sub> ... (16%)			32.2 SiO <sub>2</sub> ,CaO... (27%)							
120.2			120.2							
V pyroxenite = $\frac{101}{3.23}$			V phlogopitite = $\frac{8}{3.32} + \frac{54}{2.78} + \frac{26}{2.72}$				calculated composition of phlogopitite ( wt-% )			
			= 31.3				61 % phlogopite ρ = 2.78			
			= 31.4				30 % calcite ρ = 2.72			
							9 % diopside ρ = 3.32			
ρ pyroxenite = 3.23										

Considering the specific densities of pyroxenite, fassaite, phlogopite and calcite, and keeping the volumes of educt and product constant, the modal composition of phlogopitite can be calculated.

Eventually cataclastic or mylonitic zones are created, today replaced by phlogopitites.

- In a later phase—possibly during the panafrican orogenesis 650 m.a. ago (VACHETTE et al., 1977)—the zones of increased mobility are metasomatically transformed, the event being probably connected with the appearance of pegmatites striking discordantly through the pyroxenites in Ampandandrave and Sakamasy.
- Since certain parts of educt (pyroxenite chemistry) and product (phlogopite, diopside, calcite and anhydrite chemistry) are known, a balance calculation seems possible. This calculation is based upon averaged pyroxenite and diopside compositions from Ampandandrave and Sakamasy respectively (Joo, 1972), and upon new data on phlogopite. The chemical/mineralogical variation of the pyroxenite bodies being large, an average does not necessarily represent the truest basis for a calculation procedure and is possibly an unavoidable source of error.
- An estimation of the phlogopite mineral mode in-situ, at the mining site, is extremely difficult due the coarse and inhomogeneous character of the mined pockets. The exact proportions of the mineral present being virtually unknown, an estimation is nevertheless possible:

phlogopitite Ampandandrave    phlogopite » calcite > diopside, anhydrite  
                                      Sakamasy                    phlogopite » diopside, calcite

Based upon this indication, and further on the assumption that the volume of educt and product had to remain constant, an estimation of the mass balance involved in the derivation of phlogopitite from pyroxenite enables the following deductions, wt-% (see tables V, VI data determined by iterative calculation):

		<i>Ampandan- drave</i>	<i>Sakamasy</i>	
educt	pyroxenite	82	84	} 100%
	metasomatic supply	18	16	
	supply Al <sub>2</sub> O <sub>3</sub>	3.5	0	} % of total mass
	MgO	0	1.5	
	K <sub>2</sub> O	4.5	4	
	H <sub>2</sub> O	< 1	-	
	F	< 1	1	
	CO <sub>2</sub>	6	9.5	
SO <sub>3</sub>	2.5	0		
product	phlogopitite	71	73	} 100%
	eluate	29	27	
	phlogopitite	68	61	} 100%
	phlogopite	6	9	
	diopside	20	30	
	calcite	6	-	
	anhydrite			
eluate SiO <sub>2</sub>	18	18	} % of total mass	
CaO	10	8		
Na <sub>2</sub> O	< 1	< 1		
volume change	educt/product	< 0.5	< 0.5	(%)

By means of a metasomatic supply relatively small amounts of  $K_2O$ ,  $MgO$ ,  $\pm Al_2O_3$  and volatiles have to be introduced. This metasomatic event may be connected with the intrusion of discordant pegmatites present at Ampandan-drave and Sakamasy as well.

- The correlations of Al-rich pyroxenites delivering the majority of the essential elements for phlogopite formation, and the intrusion of pegmatitic bodies may explain why coarse grained phlogopites are worldwide scarce. A further limiting factor is certainly the necessary supply of fluorine acting as a solvent, and being typical for all known coarse grained phlogopites all over the world (Canada, Ceylon-Sri Lanka, USSR / DUNN, 1961, RIMŠAITE, 1970, SKOW, 1962).

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