

Study on the effects of doped potassium and rubidium atoms on lattice and Tc value of C₆₀ crystals

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Evolution and theory of C₆₀

Carbon is a tetravalent atom. For a long time there was a misconception that carbon only had two allotropes: diamond and graphite. However, in 1985, a group of scientists at Rice University identified a new molecule that would revolutionize organic chemistry as we know it¹. This molecule was named Buckminsterfullerene because it was a truncated icosahedron, one of the most famous geodesic domes of Buckminster Fuller, a famous American architect. Commonly called buckyball or C₆₀, this little wonder was only synthesized in sufficient quantity to prove its existence, by X-Ray diffraction, in 1990 by Kratschmer and Huffman². This experiment and others that followed not only showed that there was more than two structural possibilities for carbon, they also introduced a new family of closed caged carbons: the fullerenes.

The genius of C₆₀ said Richard Haddon is the creation of an environment in which the formation of other structural possibilities was competitive with graphite sheets¹²³. The two known techniques to produce C₆₀, graphite sublimation by laser or electric arc, both involve an inert gas environment. Rapid condensation of the carbon vapours in an inert atmosphere produces a spheroidal structure, and it is an arresting feature that is the source of the amazing electric properties of C₆₀. This perfect C₆₀ sphere can only be obtained if all 12 five-member rings are surrounded by 5 benzene rings and if a change in the angle occurs within these benzene rings. Variations in angle within the benzene rings change the hybridization of the carbon atoms. We no longer have a pure sp² hybridization but a mixture of sp² and sp³. This implies that the p-orbitals will also be impure and some s-orbital will be mixed with the p-orbital. The rehybridized (sp²)-orbitals containing the p-orbital and some lower s-orbitals benefit from an enhanced electron affinity (estimated at 2.65

eV) that makes it the most electronegative hydrocarbon³.

Figure 1

Presentation of octahedral site K(O) and tetrahedral site K(T) of C₆₀ crystal.

The strength of its electronegativity and its crystal structure are favourable for superconduction. Being a face centered cubic (f.c.c.) crystal, the C₆₀ crystal possesses three interstitial sites: one octahedral (2.07 Å radius) and two tetrahedral (1.12 Å radius) (see figure 1). These spaces are big enough to accommodate metallic ions that can transform the C₆₀ crystal into an organometallic substance without disturbing the overlap or the contact between the C₆₀ molecules.

Goal and hypothesis

Based on the experimental protocol and results of the article Structure and properties of alkali-doped C₆₀⁶ published in Material Science and Engineering 1993, B19, 135-140, this extended essay wants to demonstrate that the chemical properties of the doped alkalis influence the site of doping in the C₆₀ crystal. It will also attempt to show that the lattice value of the molecular crystal and the critical temperature (T_c) of the subsequent superconductor are also influenced by the chemical properties of the alkali. These goals will be reached using a powder X-Ray diffraction analysis and a SQID analysis of three alkali-doped C₆₀ superconductors (K₃C₆₀, K₂RbC₆₀, Rb₃C₆₀). The alkalis used to produce the C₆₀ superconductors will be potassium

1 Kroto, H.W.; Heath, J.R.; O'Brien, S.C.; Curl R.F.; Smalley, R.E.; **Nature**, 1985, 162-164.

2 Kratschmer, W.; Lamb, L.D.; Fostiropoulos, K.; Huffman, D.R.; **Nature**, 1990, 347, 354-358.

3 Haddon, R.C.; **Acc. Chem. Res.**, 1992, 25, 127-133.

and rubidium because they produce simple f.c.c. crystal when doped alone and also when doped together.

The relation between lattice value and T_c has been known for quite some time. My experimental results should therefore be representative of this relationship. This means that for each higher stable lattice value I should have a higher transition temperature (T_c). My results should also demonstrate that the bigger the alkali in weight and in mass, the higher the lattice value of the new crystal.

The interstitial position occupied by the alkali metal should be influenced by the size and the electronegativity of the metals. I would therefore expect that K_3C_{60} the potassium atoms would be evenly spread out in the spaces, as would the rubidium atoms of Rb_3C_{60} . As for K_2RbC_{60} , I suspect that Rb atoms will prefer the octahedral sites because they provide more room. This would leave the two tetrahedral sites for the smaller and less electronegative K atoms.

Description of material and manipulations

1. Production of K_3C_{60} , K_2RbC_{60} , Rb_3C_{60} ¹

In an aragon glove box, in three identified and dried glass vials, weigh 0.4000g of pure C_{60} , and add the stoichiometric quantity of the necessary alkali metals² (Figure 2).

Transfer the mixture to the homemade glass tubes (Figure 3) that have been washed in ethanol, dried and sealed.

Put 600 Torr of helium in each test tube³, (Figure 3). Heat in a furnace for 12h at 403K and for 3 weeks at 600K.

Test for air stability of compounds and wash carefully the instrument⁴.

2. SQUID analysis⁵

	Weight of C_{60}	Weight of K	Weight of Rb	Weight vials	Error
K_3C_{60}	0.4000 g	0.0651 g	0.0000 g	12.8690 g	+ ou - 0.0001 g
K_2RbC_{60}	0.4000 g	0.0434 g	0.0474 g	13.0039 g	+ ou - 0.0001 g
Rb_3C_{60}	0.4000 g	0.0000 g	0.1424 g	12.9151 g	+ ou - 0.0001 g

Means

The experiment will be separated into three steps:

1. production of the three compounds;
2. superconducting quantum interference device (SQUID) analysis; and
3. powder X-Ray diffraction analysis.

Fill a small plastic container with one of the compounds and compact the powder with white cotton.

A SQUID of quantum design MPMS was cooled down to 5K under zero Oe magnetic field.

SQUID response was evaluated.

Susceptibility ($emu\ g^{-1}\ Oe^{-1}$) was measured as a function of temperature up to 50 degrees Kelvin.

1 Tanigaki, K.; Structure and Properties of Alkali-Doped C_{60} , Material Science & Engineering, 1993, B(19), 135-140.

2 Note: One should cut off the oxidized crust of the metal and only keep the shiny metal surface.

3 The helium pressure is there for security reasons. With the heat the pressure inside the test tubes augments and prevents any entrance of air.

4 The apparatus used with K can be washed with pure ethanol in air but the apparatus used with Rb have to be washed in the glove box with a 95% Xylene and 5% butanol solution. This can easily be explained by the greater reactivity of rubidium.

The imposed magnetic field was Oe for Rb_3C_{60} et $\text{K}_2\text{RbC}_{60}$ and 20 Oe for K_3C_{60} because it had a weak signal.

3. Powder X-ray diffraction intensities

Unlike that used in the above scientific Literature, our machine did not produce X-ray with copper crystal but with cobalt. Therefore, the stopping angle had to be adjusted to the Cobalt wavelength (1.78897 Å) using Braggs law.

Figure 3

Presents the glass test tube used. Showing : A the cap, in B the joint, in C the gas entry and in D the reaction chamber.

The scan was made from the starting angle of 10 degrees to the finishing angle of 65 degrees. Each powder diffraction pattern was collected at room temperature in the 0-20 mode at a full scale of 500 and a sampling rate of 0.05 degrees per minute. The diffractometer operated at 30 kV and 20 mA. The same monochromator that was reported in the literature was placed in front of the detector.

Experimental results

1- Table of the experimental weights of C_{60} , K, Rb and He pressure

	Weight of C_{60}	Error + ou -	Weights of K	Error + ou -	Weight of Rb	Error + ou -	He pressure	Error + ou -
K_3C_{60}	0.3999 g	0.0002 g	0.0652 g	0.0002 g	0.0000 g	0.0002 g	600.1 Torr	3 Torr
$\text{K}_2\text{RbC}_{60}$	0.3999 g	0.0002 g	0.0434 g	0.0002 g	0.0474 g	0.0002 g	600.1 Torr	3 Torr
Rb_3C_{60}	0.4000 g	0.0002 g	0.0000 g	0.0002 g	0.1423 g	0.0002 g	599.8 Torr	3 Torr

2- Table of the percentage loss of K and Rb during transfers to test tube

	% loss of K	Error + ou -	% loss of Rb	Error + ou -	Total % loss	Error + ou -
K_3C_{60}	5.2147%	0.3325%			5.2147%	0.3225%
$\text{K}_2\text{RbC}_{60}$	3.4562%	0.4768%	2.7426%	0.4335%	6.1988%	0.9103%
Rb_3C_{60}			3.7948%	0.1458%	3.7948%	0.1458%

3. Graphic results from the SQUID analysis

Graph 1 presents the SQUID's response to the K_3C_{60} sample. This shows the relationship between the imposed 20 Oe magnetic field on the sample and the voltage in the SQUID circuit.

Graph 1

Graph 2 presents the relationship between the temperature (in Kelvins) and the Susceptibility ($\text{emu} \times \text{g}^{-1} \times \text{Oe}^{-1}$) for the $\text{K}_2\text{RbC}_{60}$ compound.

Graph 2

x versus temperature of $\text{K}_2\text{Rb}_1\text{C}_{60}$

Graph 3 presents the same relationship as graph 2 but for the Rb_3C_{60} compound.

Graph 3

x versus temperature of Rb_3C_{60}

Table A presents the transition temperature (T_c) of the three compounds, It is important to note that the precise identification of the temperatures was made mechanically and that there was an error of + or - 1K made by the machine.

Table A:

	T_c	Error + or -
K_3C_{60}	none	
$\text{K}_2\text{RbC}_{60}$	21K	1.5 K
Rb_3C_{60}	29K	1.5 K

4- Graphic results from the powder X-ray diffraction analysis

This section presents the powder X-ray diffraction graphs. All graphs present the relation between the refraction angle of the initial X-ray wave, after intercepting the samples, versus the intensity of these refracted X-rays.

Discussion

a) The SQUID analysis

The SQUID is very sensitive to variations in magnetic fields. Its operations are based on the quantization of the magnetic flux and on the Josephson effect. For the purpose of this study, the understanding of the Josephson junction is very important in order to understand the SQUID response presented in Graph 1.

Figure 4 presents the Josephson junction. The insulating sheet between the two superconductors is only a few nanometres thick. If the material is not superconducting, the electrons travelling through the circuit would be stopped by the insulating sheet and a voltage would appear. In superconductors, it seems that the Cooper pairs can pass through the insulating sheet by a tunnelling effect. For low current density, it has been shown that the insulating sheet becomes superconducting. In a closed circuit, there should theoretically be no voltage across the Josephson junction.

Figure 4

A SQUID response (as presented in Graph 1), based on the Josephson effect, evaluates whether the material under analysis is a superconductor. For Rb_3C_{60} and $\text{K}_2\text{RbC}_{60}$, the SQUID response was negative. This meant that there was no voltage in the Josephson junction and that the two materials were superconductors. As we can see in Graph 1, K_3C_{60} presented a voltage. This meant that this compound was not a superconductor. The causes for this will briefly be explained in another section.

Theoretically, the graph presenting the relationship of temperature (K) versus the susceptibility should look like the graph in Figure 5.

Figure 5

The transition temperature (T_c) from normal conductivity to superconductivity is very easy to identify on figure 5. For an infinitely steep slope, the T_c represents the point of the graph just underneath the temperature axis.

In the experimental graph (2 & 3), we see that the experimental slopes are not as steep as the theoretical slope. This implies that the transition phase from normal conductivity to superconductivity is not as well defined as in the theoretical graph. A good superconductor is a material whose transition phase is isolated to one temperature.

Because the disordered phase in graphs 2 & 3 are extended to more than one possible temperature, it is reasonable to assume that the two compounds are relatively weak superconductors. A long transition phase makes it more difficult to precisely identify the critical temperature of the compound. The critical temperatures of K_2RbC_{60} and Rb_3C_{60} as presented in table. A were given a large margin of error because of the uncertainty in the mechanical identification of the temperature and because of the uncertainty in the exact value of the T_c .

Most of the referenced papers used in this extended essay make no mention of margin of errors. It is therefore very difficult to present tests to prove that the experimental results of this paper are compatible with the results of other scientists. If I were to take the values presented in the article from Material & Engineering for the real values of T_c for the three superconductors, only Rb_3C_{60} would concur with their results. Taking into account that my T_c seemed to be lower than theirs, this difference in T_c value can be explained by the fact that my superconductors are extremely reactive, it is possible that the contact with air might have led to the

degradation of the superconducting properties¹. On the other hand, if compared with the results of papers² that include a margin of error, my result concur with theirs. Table B summarizes.

Table B:

	Experimental T_c	Reference T_c	Other T_c
K_3C_{60}	None	19 K	18.0-19 K
K_2RbC_{60}	21 K + or - 1.5 K	23 K	21.8-22.5 K
Rb_3C_{60}	29 K + or - 1.5 K	29 K	28.0-29.8 K

The above results clearly show that there is a relationship between the size of the doped atoms the higher the critical temperature. As we can, the bigger the doped atoms the higher the critical temperature of the superconductor. Naturally, one must admit that the sampling is not extensive and that this hypothesis is based on two values. Nevertheless if this hypothesis is compared with the results of others, we can see that an almost linear relationship has also been reported in numerous papers.

1 Arthur P. Ramirez, **C60 And Its Superconductivity**, Superconducting Review 1994, Vol 1, p. 1-101.

2 A) Holzzer, K.; Klein, O.; Huang, S.-M.; Kanner, R.B.; Fu, K.J.; Whetten, R.L.; Diederich, F.; **Science** **1991**, 252, 1154.

B) Fleming, R.M.; Ramirez, A.P.; Rosseinsky, M.J.; Murphy, D.W.; Haddon, R.C.; Zahurak, S.M.; Makhija, A.V.; **Nature** **1991**, 352, 787.

b) *Powder X-ray diffraction analysis*

As shown by the German physicist Von Laue in 1912, we know that in a crystalline structure traversed by X-rays a constructive interference occurs within the crystal. This interference splits the X-ray beam into multiple components that are reflected in different directions¹. Later, Bragg proved that Von Laue's hypothesis was well-founded if you considered that the X-rays were reflected by sets of parallel planes for very specific angles. He found that the sine of that angle had to equal the ratio between the wavelength of the X-rays and the double of a constant: d . This constant d is very important for the purpose of this experiment because it will enable us to calculate the size of the unit cell of a particular crystal.

The peaks on the powder X-ray diffraction graphs can be considered as local intensity maxima. Basically, the peaks represent a position within the crystal where the reflected waves are in phase. Using the raw data, the theta angle corresponding to these local intensity maxima were isolated. Table 1, 4, 7 outline the theta values isolated for each compound. They also present the lowest and highest theta value that are acceptable within the margin of error.

1 Theodore Kuwana, **Physical methods in modern chemical analysis**, AP, 1980, Volume 2.

Table 1:

K_3C_{60}	Intensity	Number	Theta value	Error + or -	Low value	High value	Plan Coording
01	155	84	6.125 deg	0.125 deg	6.000 deg	6.250 deg	(1,1,1)
02	15	129	7.260 deg	0.125 deg	7.135 deg	7.385 deg	(2,0,0)
03	230	245	10.125 deg	0.125 deg	10.025 deg	10.275 deg	(2,2,0)
04	178	316	11.925 deg	0.125 deg	11.800 deg	12.050 deg	(3,1,1)
05	49	339	12.500 deg	0.125 deg	12.375 deg	12.625 deg	(2,2,2)

Table 4:

K_2RbC_{60}	Intensity	Number	Theta value	Error + or -	Low value	High value	Plan Coording
06	67	84	6.15 deg	0.125 deg	6.000 deg	6.250 deg	(1,1,1)
07	0	0	0 deg	0 deg	0 deg	0 deg	(2,0,0)
08	78	245	10.150 deg	0.125 deg	10.025 deg	10.275 deg	(2,2,0)
08	102	317	11.950 deg	0.125 deg	11.825 deg	12.075 deg	(3,1,1)
10	29	340	12.525 deg	0.125 deg	12.400 deg	12.650 deg	(2,2,2)

Table 7:

Rb_3C_{60}	Intensity	Number	Theta value	Error + or -	Low value	High value	Plan Coording
0a	77	86	3.175 deg	0.125 deg	6.050 deg	6.300 deg	(1,1,1)
0b	14	129	7.250 deg	0.125 deg	7.125 deg	7.375 deg	(2,0,0)
0c	158	246	10.175 deg	0.125 deg	10.050 deg	10.300 deg	(2,2,0)
0d	114	317	11.950 deg	0.125 deg	11.825 deg	12.075 deg	(3,1,1)
0e	39	339	12.500 deg	0.125 deg	12.375 deg	12.625 deg	(2,2,2)

The angle values in the above Tables are the angles at which constructive interference occurs within the crystals. Using these angle values, one can find the Bragg's constant D.

For example:

Bragg's law says that $A = 2D\sin\theta$

Where $A = \text{Wavelength (A)}$

$\theta = \text{The angle (degrees)}$

$D = \text{The ratio (constant)}$

We know that $A = 1.79021A$

$\theta = \text{The values in Table 1}$

If $A = D$, we find that $\frac{1.79021A}{2\sin\theta} = 8.389 A = D$

D is constant and particular to each peak. Always using Bragg's law, the Bragg's experimental ratio (Dm) for all five peaks was calculated. The lowest (DI) and highest (Dh) ratios possible for each peak, within the margin of error of + or - 0.123 deg. per theta angle were also calculated. The resulting ratios for each crystal are presented in Tables 2,5,8:

Table 2: (Unit in A)

K_3C_{60}	DI	Dm	Dh
01	8.563	8.389	8.222
02	7.207	7.083	6.964
03	5.142	5.079	5.018
04	4.377	4.332	4.288
05	4.177	4.136	4.095

Table 5: (Unit in A)

K_2RbC_{60}	DI	Dm	Dh
06	8.563	8.389	8.222
08	5.142	5.079	5.018
09	4.368	4.323	4.279

10	4.168	4.127	4.087
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Table 8: (Unit in A)

Rb_3C_{60}	DI	Dm	Dh
0a	8.493	8.322	8.157
0b	7.217	7.093	6.973
0c	5.129	5.067	5.006
0d	4.368	4.323	4.279
0e	4.177	4.136	4.095

The mentioned D constant and the Cartesian coordinate known, the calculation of the lattice parameter should become easier. We must however explain what these coordinates are and what they represent. The coordinates are technically named Miller's indices and are graphically represented by X-ray diffraction lines. They are a special notation made to identify the sets of parallel planes inside the crystals. Their value is estimated by the position occupied by the plane in reference to axes XYZ located at the one of the corner of the f.c.c. cube. The planes contain the most lattice points (the centre of the atoms in the crystal) possible. In order to yield reflection, the value of the Miller's indices for f.c.c. must all be even or odd¹.

The experimental graphs were given indices as shown in Tables 1,4,7 that would serve with the calculated D values to find the lattice parameter using the formula for Interplanar Spacing for a f.c.c.

The formula of Interplanar Spacing for f.c.c. says that:

$$D_2 = \frac{a^2}{h^2+k^2+l^2}$$

¹ The repartition of Miller's indices for each peak must assure that all lattice values calculated are equal (within the acceptable margin of error).

Where D = The ratio (cst)
 a = The lattice value
 h, k, l = The Miller indices of a peak
 If we take our previous example we would find that:
 $(8.389 \text{ \AA})^2 \times (1^2 + 1^2 + 1^2) = 14.530 \text{ \AA} = a$

The lattice parameter is constant from all peaks for the same crystal. I therefore calculated the average lattice parameter of all the experimental lattices (am) and the average lattice parameter for the lowest (al) and highest (ah) lattices possible for the significative peaks. Table 3,6,9 summarize the lattice values found for each and the average lattice value for K_3C_{60} .

Table 3: (Unit in A)

K_3C_{60}	al	am	ah
01	14.832	14.530	14.240
02	14.414	14.166	13.928
03	14.544	14.366	14.193
04	14.517	14.368	14.222
05	14.470	14.328	14.186
Average	14.555	14.352	14.154

Table 6: (Unit in A)

K_2RbC_{60}	Al	Am	Ah
06	14.832	14.530	14.240
08	14.544	14.366	14.193
09	14.487	14.338	14.192
010	14.487	14.296	14.158
Average	14.575	14.383	14.196

Table 6: (Unit in A)

Rb_3C_{60}	Al	Am	Ah
0a	14.710	14.414	14.128
0b	14.434	14.186	13.946
0c	14.507	14.332	14.159
0d	14.487	14.338	14.192
0e	14.470	14.328	14.186
Average	14.522	14.319	14.122

As we can see by comparing the experimental results with the reference results, the proposed lattice value in the literature is included in the acceptable interval of calculated values for this experiment. However, if we only look at the average lattice values for the three crystals, we realize that Rb_3C_{60} which supposedly possessed the biggest unit cell, has the smallest unit cell of the three crystals. If this were true, it would imply that the two starting hypotheses concerning the direct relationship between the size of the doped atoms, the size of the unit cell and the length of the transition temperature would not concur with the results of other papers on this subject. But considering that the margin of error is acceptable and that the first hypothesis concerning the relationship between the size of the alkali and the critical temperature is valid, we have to admit that, within the experimental interval of lattice values possible, the observed growth in lattice parameter with the augmentation of metal size is feasible (even if not directly observed for all three crystals).

One should also realize that there is a second phase, an impurity, found in all three samples. The unidentifiable peaks in the 30 deg-40deg (20) regions as well as the width of the diffraction peaks are proof of the presence of this impurity in the crystal's structure. It is reasonable to assume that the presence of this impurity in the crystal might have been the cause of the none superconducting state of K_3C_{60} . The presence of this impurity might also have been responsible, beside the air exposure time, to the lowering of the transition temperature for the K_2RbC_{60} .

Impurities in C_{60} crystals are not uncommon. Their presence and other factors make it difficult to produce good single crystals. The rarity of good single crystals is an important obstacle to single crystal X-ray analysis which, as we know, is the best way of determining the position of atoms in a crystal. For powder X-ray diffraction, however, the presence of single crystals is not necessary. This analysis does not provide as much information concerning the precise position of the atoms in the crystal structure as the single X-ray diffraction analysis, but it can reveal important information concerning the similarities between different crystals and their unit cell value. There are alternatives using simulated profiles of powder X-ray patterns for particular atomic configuration (as used in the above literature) that can identify the exact atomic structure of the crystal. Unfortunately, these alternatives could not be exploited for the purpose of this experiment because the proposed simulations were for a binary C_{60} superconductor (Na_2CsC_{60}). There are numerous similarities between K_2RbC_{60} and Na_2CsC_{60} such as the ratio between alkali atoms and the class of crystal (f.c.c.), but it does not seem appropriate to compare the K_2RbC_{60} X-ray graph with the simulations prepared for Na_2CsC_{60} .

Without the use of simulation graphs for various atomic arrangements, it is not possible to verify completely our third hypothesis concerning the position of the alkali metals in the crystal. This means that although it is very logical that the Rb atoms, being bigger and more positively charged than potassium, should be doped in the octahedral sites, we cannot prove this hypothesis with the present experimental results. It can however be said that whatever the structure of the crystals, K_3C_{60} and Rb_3C_{60} have a very similar structure. Looking at their X-ray diffraction pattern, we clearly see that the significant peaks are the same and that their intensities seem to be of the same order. We can also say that K_2RbC_{60} differs in structure from the two other crystals because there is no (2,0,0) peak and because the ratios between each peak's intensities does not concur with the ratios of the two other crystals. In the K_2RbC_{60} graph for example, the peak (2,2,0) is lower than peak (3,1,1). In the two other crystals, the peak (3,1,1) was the lowest.

Conclusion

In this extended essay, three doped C_{60} crystals were studied: K_3C_{60} , K_2RbC_{60} and Rb_3C_{60} . Based on SQUID analysis and powder X-ray diffraction analysis we have established that the three doped crystals are face centred cubic crystals (f.c.c.) and two of the three compounds possess superconducting properties. It has also been shown that a direct relationship exists between the size of the doped alkali and the critical temperature at which there is a transition from normal conductivity to superconductivity. Within the margin of error, another direct relationship between the alkali size and the value of the unit cell is strongly suggested. Many resemblances between K_3C_{60} and Rb_3C_{60} have been revealed by X-ray diffraction patterns, but no precise atomic structure of the two crystals or precise explanation for the variation of structure for K_2RbC_{60} can be offered with the present experimental data. These conclusions, because of the limited sampling, are reasonable if compared with the mentioned experimental findings.

Acknowledgments

The author is grateful to Dr. John Morton, Dr. Gary Enright and Mr. Régent Dutrisac of the Steacie Institute for Molecular Sciences (SIMS) at the National Research Council (NRC) for both their technical and moral support. Special thanks to Dr. Julian Cave of IREQ, Dr. Tapan Boze and Dr. Ravi Gopal of Québec University at Trois-Rivières (UQTR) for their technical assistance. A Special mention to M. Alain Staub, M. Bernard Dugas and M. Maurice Hébert of Brébeuf College for all their financial and moral support.

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