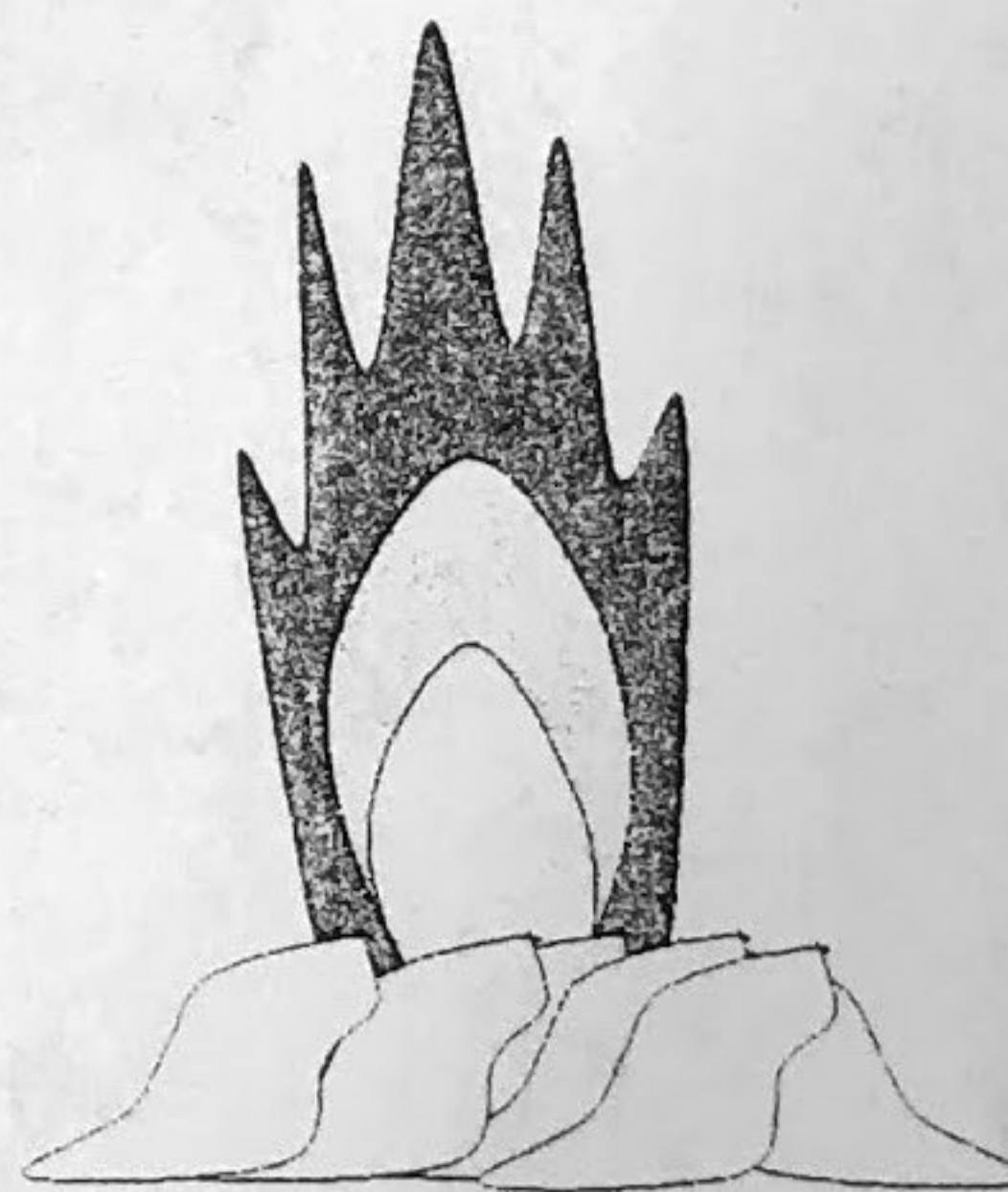
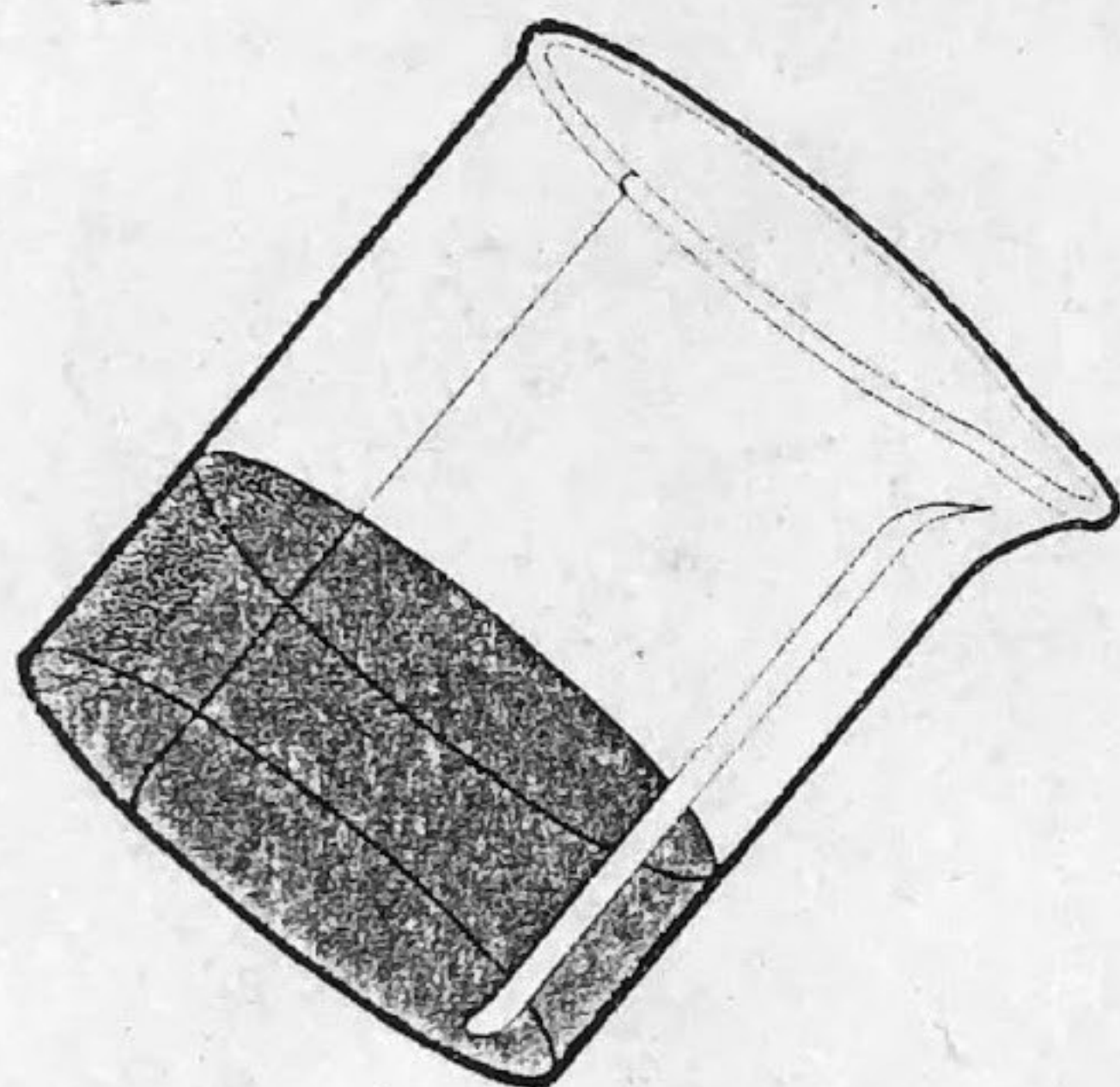


Test for Spontaneous Combustion of Biodiesel

*Idaho
Bioenergy Program*



Idaho Department of Water Resources

 *Energy Division*

March 1996

**Test for Spontaneous Combustion
of Rapeseed Biodiesel**

by

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INTRODUCTION

Self-heating is the occurrence of a rise in temperature in a body of material in which heat is being generated by some process taking place within the material. Ignition hazards that arise from self-heating are not uncommon in the process industries. Self-heating may be inherent in the system due to the storage and/or processing of a material during which exothermic reaction occurs. Storage of grain, hay, foodstuffs, coal, oil soaked rags and sawdust have all been involved in loss-producing situations. Reaction is initiated by the high temperature and the exotherm may be sufficient to cause smoldering and ignition. Self-heating may be influenced by the presence of water in liquid form or moisture in the atmosphere.

Napier and Vlatis (1982) reported that seed residues, after crushing are usually mixed with other materials and used as cattle feed. The possibility arises that the prolonged storage of this mixture may present a hazard. Seven firms involved in seed crushing were surveyed and most of the firms had not encountered severe hazard during storage. One firm had noted that with high moisture content, hazards are possible. A second firm reported that a fire had started in the storage area subsequent to considerable leakage of water into the area. The assessment of frequency of a hazardous situation arising was about three events in 100 years.

Spontaneous combustion is possible with any oxygenated fuel or oil if care is not taken. Many factors are involved for spontaneous combustion to take place i.e., piles of sawdust, wind, various levels of relative humidity, water, a catalyst such as steel, heat generated by electric motors, etc.

Biodiesel from rapeseed oil as an alternative fuel has been under study at the University of Idaho since 1979. To produce Biodiesel, the rapeseed oil is transesterified using either ethanol or methanol with potassium hydroxide as a catalyst. The resulting ester has very good potential as a diesel fuel substitute. This paper examines the potential of the biodiesel to self ignite from spontaneous combustion when rags, paper or other material soaked with the biodiesel are left in a pile or uncovered receptacle.

REVIEW OF LITERATURE

Bowes (1984) reports on a fire involving 400 tons of bagged rape seed and believed to have been due to self-ignition. Self-heating had been observed some weeks before the fire; temperatures of 50°C (122°F) and 73°C (163°F) were measured and smoke haze and decomposition products (said to be acrolein) were present in the atmosphere, causing headaches and eye irritation. Heated bags were removed and empty bags were placed in a heap. It was later thought, however, that some damaged bags of seed had been left in the pile. When fire broke out later, damage was extensive and it was uncertain whether it had originated in the seed or in the heap of empty bags. The ability of the empty bags to self-heat was investigated when a temperature of 89°C (192°F) was observed accompanied by a

bluish acrid smoke. The store also contained 1500 tons of wheat in bags and 50,000 empty bags which had contained wheat. There was no evidence of heating in the wheat.

Bowes (1984) also reports critical ignition data for rape seed in cube-shaped containers, ranging in size from 50 mm to 200 mm. Self-heating to temperatures in excess of 200°C (392°F) or so was accompanied by congealing of the seeds into a charred oil-soaked mass. Identification of a critical temperature was subject to some uncertainty but, adopting the final appearance of ash as the criterion of ignition, the critical data for the rape seed, which had an initial moisture content of 7.2% and an oil content of 36.9%, was represented by the following equation (with a linear correlation coefficient of unity to three significant decimals):

$$\ln \frac{\delta_c T_A^2}{r^2} = 22.6091 - \frac{8519}{T_A}$$

where r is the radius or half thickness in mm and T_A is in degrees Kelvin.

Bowes (1984) concludes that at an ambient temperature of 293°K (68°F), the above equation indicates a critical size of $(2r)$ of 24 m for self ignition of a cube-shaped pile of dry seeds. They suggest that the practical risk of self-ignition in rape seed is negligible. However, assuming that the simple model for parallel reactions can be applied, and neglecting the effects of moisture on thermal properties, it may be estimated that the fire could have been spontaneous in origin.

The Condensed Chemical Dictionary reports the following properties for soybean oil, rapeseed oil and linseed oil:

Soybean oil: Saponification value 190-193, iodine value 137-143, flash point 540°F, combustible, nontoxic. Moderate spontaneous heating. Autoignition temperature 833°F.

Rapeseed oil: Flash point 325°F, autoignition temperature 836°F, subject to spontaneous heating. The iodine value is 97 for industrial rapeseed.

Linseed oil: Saponification value 189-195, iodine value 177, flash point 432°F, autoignition temperature 650°F. Heats spontaneously; combustible; nontoxic.

A material safety data sheet for boiled linseed oil from Startex Chemical, Inc. (Anon, 1987), states under section V: Reactivity data that spontaneous combustion can occur; and under section IV, that rags and waste paper containing this material may heat and burn spontaneously.

DEFINITIONS

Autoignition temperature - The lowest temperature at which the chemical reaction proceeds at a rate sufficient to result eventually in inflammation. Subjecting a mixture to a temperature higher than the autoignition temperature results in inflammation after a short time lag. There is a temperature for each material which results in practically instantaneous inflammation.

Flash Point - The lowest temperature to which a fuel must be heated to produce an ignitable vapor-air mixture above the liquid fuel when exposed to an open flame. At temperatures below the flash point, not enough fuel evaporates to form a combustible mixture.

Iodine Value - A number expressing the percentage of iodine absorbed by a substance. It is a measure of the unsaturation level of an animal fat or vegetable oil.

Saponification Value - The milligrams of potassium hydroxide required for the hydrolysis of an animal fat or vegetable oil into glycerol or soap. It is useful in providing information relative to the proportion of glyceride and fatty acids present in a sample.

Self-heating - The occurrence of a rise in temperature in a body of material in which heat is being generated by some process taking place within the material.

Spontaneous Combustion - Inflammation of a material due to self-heating sufficient to reach the autoignition temperature.

EMPIRICAL METHODS OF ASSESSMENT

The following three methods have been developed to assess the spontaneous combustion capabilities of materials.

1. Standard Test Method for Spontaneous Heating Values of Liquids and Solids (Differential Mackey Test, ASTM D 3523-86)

This test method covers the non-adiabatic determination of the spontaneous heating values of liquids and solids. Spontaneous heating values obtained by this test method are qualitative indications of the degree of self-heating that may be expected to occur upon exposure of the sample to air at the test temperature. With a sufficiently reactive oil, a pile of oiled and blended wool could self-heat to charring temperature and even active combustion.

In this test, the sample is supported on surgical gauze and placed in a heated chamber which is open to the air at the top. The temperature, of a saturated 20g of freshly rolled cotton gauze with 10g of liquid, is compared to that of clean surgical gauze

contained in an identical chamber. The amount by which the temperature of the sample exceeds that of an equal reference quantity of surgical gauze is taken as an index of the spontaneous heating value of the sample. Tests may be conducted for durations of 4 to 72 hours or longer if desired.

A weakness of the test, which eventually became apparent, was the time limit on the specified temperature rise. The addition of anti-oxidant could extend the induction period for oxidation sufficiently to enable an otherwise "unsafe" oil to pass the test. While the anti-oxidant could, at the same time, provide protection from heating in piles of blended wool at ordinary temperatures, it would not necessarily do so if the piles were left undisturbed for much longer than usual.

2. **Standard Test Method for Spontaneous Heating Tendency of Materials (ASTM E 771-90)**

Summary:

A small quantity of the test material held within a closely packed inert material is heated in a thermostatically controlled chamber and the sample temperature is monitored to determine the temperature rise due to exothermic reaction. Bath temperature, sample quantity, and particle sizes of solids are varied to obtain the relative self-heating temperature of the material.

Significance and use:

This test method is applicable to solid and liquid materials and provides a means of accelerating the tendency of a material toward spontaneous heating which may eventually lead to a fire.

Tests at temperatures covering the range expected in manufacturing process or material usage can be of considerable value in determining safe operating conditions.

Exothermic reaction under test conditions is a positive indication of spontaneous heating tendencies of a material. Negative test results indicate the absence of detectable spontaneous heating behavior under the experimental conditions, but should not be regarded as conclusive for all conditions, particularly those which may be considered adiabatic.

The spontaneous heating behavior of a material is affected by such factors as available surface area, availability of oxygen to the test specimen, humidity, sample moisture content, packing density, the test temperature, and loss of exothermic heat to the surrounding.

Apparatus:

The complete apparatus consists of a test chamber with a sample well and auxiliary equipment including a heater and temperature control system, supplementary

electronic bath temperature controller, high temperature limit switch, motor stirrer, multichannel recorder, thermocouples, and controlled air supply.

Materials:

The heat-transfer medium shall be any suitable oil with a flash point well in excess of the maximum operating temperature of 250°C (482°F). The flash point of the oil should be checked periodically as it may decrease with use.

Borosilicate wool filtering fiber is used as the outer packing in the sample holder.

An inert fiber is used as the inner packing in the sample holder as a liquid absorbent of powder dispersant.

A 13-mesh iron or nickel cylinder 2.5 inches in diameter and 6 inches high is used to contain the prepared test sample in the sample holder. An 8 by 6 inch section of iron mesh is rolled to form a cylinder. The 6 inch edges are folded back 175 degrees to facilitate a quick interlocking closure.

Interpretation of Results:

A rapid temperature rise resulting in specimen combustion is a definite indication of spontaneous heating behavior.

A sample temperature rise of at least 5°C (9°F) from steady state is an indication of spontaneous heating tendency.

A test is considered negative when the specimen temperature remains below the bath temperature during a 24 hour test period.

3. **Spontaneous Combustion of Linseed oil in Sawdust (Ettling and Adams, 1971)**

To determine the effect of several factors on the spontaneous combustion of a linseed oil and sawdust mixture, a series of laboratory experiments were conducted. The tests were conducted in open topped, 5 gallon metal cans in a fume hood and arranged according to the scheme in Figure 1. A layer of sawdust from a carpenter's shop was put in the can and a small depression was made in the center. The mixture of experimental sawdust and oil was dumped into the depression without compressing or packing. Thermocouples were introduced, generally one near the center of the ball of sawdust and oil and one near the edge of the ball. A light covering of sawdust was sprinkled over the ball to provide thermal insulation. The thermocouples were attached to a 12-point strip chart recorder. The cans were set in a fume hood, which carried off smoke and fumes as they were formed.

The linseed oil used in all experiments was ADM Pol-Mer-Ik "boiled". The cans were unopened until oil was first used from them. Sufficient oil for the experiment

was poured and then the can was flushed with nitrogen and recapped. In some experiments, oil from an older can was used when fresh oil was not necessary. This oil had considerable exposure to air.

The sawdust for the experiments was obtained from a wood products plant and consisted of mostly Douglas fir. It contained 14.5% moisture content (d.w.b.) and had bulk densities of 0.14 g/cc (loose) and 0.22 g/cc (packed).

Copper-constantan thermocouples were used with a Brown 12-point recorder. The span was adjusted to give from about +5°C to about 500°C.

The procedure normally used for preparing the oil and sawdust mixture was to weigh the desired amount of sawdust in a large beaker then pour in the desired amount of oil. The oil and sawdust were thoroughly mixed with a spatula until the sawdust appeared uniformly coated with oil. This loose mixture was poured into the depression in the sawdust in the test can.

The sawdust-oil mixtures in the cans were allowed to stand at ambient temperature in the draft hood. When the oil began to warm, the increase in temperature was shown on the recorder. The induction period was figured as the time between when the oil and sawdust was mixed and when the temperature began to rise rapidly. There was usually a period of about two hours of a slow temperature rise of about 10 - 20°C that preceded the rapid rise. In instances where there was no rapid temperature rise, the induction period was figured as the time up to measurable temperature increase plus one hour. In the experimental data, burning refers to smoldering of the sawdust. No free flame was ever observed.

Experiment 1: Critical Mass of Sawdust and Oil

This experiment was conducted to find the smallest mass of oil and sawdust in 1:1 weight ratio that could generate sufficient heat to cause burning. The test was set up according to the general procedure given above.

Experiment 2: Dilution of Oil in Sawdust

Oil was mixed with varying amounts of sawdust to find the limit of dilution of the oil that would generate enough heat to lead to burning. The experiment was set up according to the general procedure.

Experiment 3: Effect of Temperature on the Induction Period

An experiment was conducted with the sawdust-oil (1:1) mixture held at specific temperatures in order to find the effect on the length of the induction period. The experiment was conducted in the usual manner, except that the sawdust-oil mixture was put in a smaller can, which was set in the 5 gallon can. The 5 gallon can was either heated by a hot plate or cooled by running tap water to provide a constant temperature environment for the smaller can of sawdust.

Experiment 4: Exposure of the Oil to Air

Since exposure to air is necessary to allow oxidation that generates the heat leading to spontaneous combustion, an experiment was conducted to find the effect of exposure of the oil to air prior to mixing it with the sawdust. Four beakers containing 100 g samples of oil were let stand in air at 25°C for various lengths of time. Then they were mixed with equal weights of sawdust and set up as in the general procedure.

Infrared spectra from 2.5 to 3.5 micro were run on the oil samples after the indicated exposure to air. There was no change in the spectrum, which includes the region where hydroxides or hydroperoxides would absorb light.

Experiment 5: Moisture Content of Sawdust

An experiment was conducted to find if moisture content had any influence on the induction period. Sawdust was either dried in an oven or open in the room. Another sample was moistened and let come to equilibrium in a closed container. The rest of the experiment was conducted according to the general procedure using 60 grams of sawdust and 60 grams of oil.

Experiment 6: Availability of Oxygen

Two experiments were conducted to show how the availability of oxygen influences the spontaneous heating of the oil. In the first set, the cans were arranged according to the standard procedure, except that one ball of oil plus sawdust (1:1) was covered by 10cm of sawdust, another was flush with the surface, and a third was half exposed above the bed of sawdust.

In the second experiment, a beaker was insulated and then filled with a sawdust-oil mixture (1:1). Thermocouples were placed in the center of the beaker at depths of 2.5, 5, 7.5, and 10 cm., and another was placed near the wall of the beaker at a depth of about 7.5 cm.

Experiment 7: Foreign Material

Since materials might have a catalytic effect on the oxidation of the oil, experiments were conducted with several foreign materials mixed with the oil. Otherwise, they were conducted according to the standard procedure using 60 grams each of sawdust and 60 grams of oil.

Experiment 8: Freshness of Oil

Oil from two previously unopened cans were used in an experiment to confirm that fresh oil has a long induction period. The experiment was conducted according to the standard procedure using 60 grams of sawdust and 60 grams of oil.

Experiment 9: Ignition Temperature of Sawdust

Electric heaters were used in the 5 gallon cans over a layer of sawdust. Sufficient experimental sawdust was put around the heaters to cover them with at least 2.5cm in

any direction. One thermocouple was touching the heater, and another was about 1.3cm away in the sawdust. The heater was warmed slowly to the desired temperature and held at that temperature for several hours.

Experiment 10: Take-up of Oil by Sawdust

Several experiments were conducted to determine the amount of oil that will soak into sawdust under various circumstances. A puddle of 18 grams of oil on a smooth counter top was covered with sawdust and let stand for one hour. The dry sawdust was gently blown away, leaving oil soaked sawdust. The mixture of oil and sawdust was weighed and the oil on the counter top was wiped up and weighed. The sawdust-to-oil weight ratio was 1.04:1 and only 1% of the oil was left on the counter top.

A similar experiment with a puddle of oil in a large watch glass showed a sawdust-to-oil ratio of 0.70:1.

In a third experiment, 6 grams of oil were poured into one spot in an excess of sawdust. After standing 1.5 hours, the oil soaked sawdust was removed and weighed. The sawdust-to-oil ratio was 0.86:1.

In several other experiments, oil was poured into sawdust and let soak in with sufficient time to harden (48 hours or more). The oil in 11 balls ranged from 0.36 g to 14 g. Excess sawdust was removed from the hardened balls, which were then weighed. The ratios of sawdust-to-oil ranged from 1.17:1 to 1.5:1 with a mean of 1.35:1.

Experiment 11: Critical Mass of Sawdust and Oil at Optimum Conditions

Test cans were arranged with a pocket of experimental sawdust at the surface of the scrap sawdust and level with it. Various amounts of oil were poured into each pocket and allowed to soak in without influence. Thermocouples were inserted into the oil soaked part, and then a 1.3 cm layer of dry sawdust was spread over the oiled sawdust.

Experiment 12: Effect of Depth of Cover at the Critical Mass

The test cans were arranged with a pocket of sawdust that was level with the other sawdust. A 14 gram sample of oil was poured into the sawdust and allowed to soak in. Thermocouples were inserted, and then the oil was covered by a layer of specific depth.

Other test procedures may give indication that a fluid is fire resistant or have a decreasing tendency to spontaneously combust. The flash point is the temperature at which vapors above the fluids surface ignites. The fire point is the temperature where the fluid itself ignites and burns for at least 5 seconds (ASTM D-92-66). The autoignition temperature (AIT) is determined by the fluid being injected via a syringe onto the surface of an Erlenmeyer flask heated on a hot plate. This test measures the

spontaneous ignition temperature of a fluid in air and the results are geometrically dependent (ASTM D-2155-6). The heat of combustion is determined according to ASTM D-240. As the heat of combustion decreases, the resistance to fire increases.

OBJECTIVE

To compare the spontaneous combustibility of Rape Methyl Ester (RME), Rape Ethyl Ester (REE), and boiled linseed oil.

PROCEDURE

The test procedure used was similar to that proposed by Ettlign and Adams (1971). The test was conducted in an open topped metal can. A layer of sawdust from a carpenter's shop was put in a can and a small depression was made in the center. The can was placed in an oven and the temperature was maintained at 200°C (392°F). The mixture of experimental sawdust and oil was placed into the depression without compressing or packing. A thermocouple (type T) was introduced near the center of the ball of sawdust and oil. A light covering of sawdust was sprinkled over the ball to provide thermal insulation. The thermocouple was attached to an HP Data Acquisition unit.

The procedure normally used for preparing the sawdust and oil mixture was to weigh the desired amount of sawdust in a large beaker then pour in the desired amount of oil. The sawdust and oil were thoroughly mixed with a spatula until the sawdust appeared uniformly coated with oil. This loose mixture was poured into the depression in the sawdust in the test can. The oils tested included rape methyl and rape ethyl ester and 20 percent rape methyl ester - 80 percent diesel blend and a commercial boiled linseed oil. The esters were prepared by technicians at the University of Idaho Department of Agricultural Engineering laboratory from industrial rapeseed oil from the variety Dwarf Essex which is high in erucic acid.

RESULTS

The results, shown in Figures 2 through 6, indicate the rate of temperature rise is higher in the case of linseed oil. Linseed oil is known for its spontaneous combustibility. The blend of RME and diesel fuel was different when compared with the other oils. It did not emit any smoke, and the rate of temperature rise was the least.

DISCUSSION

Bowes (1984) found that, "At ordinary atmospheric temperatures the risk of self-ignition in small quantities of fibrous materials impregnated with oils is limited to the more highly unsaturated vegetable oils of which linseed oil is the best known example." He states that the extent of unsaturation of the oils (the proportion of ethenoid bonds distributed among the constituent fatty acids) is measured by the iodine value and can provide a first indication of

the relative self-heating tendencies likely to be associated with different oils. The "drying oils" having iodine values ranging from 120-141, such as soybean oil, to 160-204, such as tung oil and linseed oil, are the most hazardous. At the other end of the scale are the "non-drying" oils and solid vegetable fats for which iodine values range from 80-88, such as olive oil, down to 8-10, such as coconut oil. Between these two groups lie the "semi-drying" oils with iodine value of 90-117, such as cottonseed oil, and iodine values of 127-136, such as sunflower-seed oil. Industrial rapeseed has an iodine value of 97-108 and canola (which is compositionally similar to soybean oil has an iodine value of 100-105 (Swern, 1979).

Bowes (1984) cautions that, "however, the iodine value is not a reliable guide to relative spontaneous combustion hazard because the rate of oxidation of the oils is governed by the distribution of the ethenoid bonds among the component fatty acids." He illustrates this by comparing the oxidation of the methyl esters of oleic, linoleic and linolenic acids which contain respectively one, two and three ethenoid bonds per molecule; the relative rates of oxidation at 20°C (68°F) are in the ratios of approximately 1:12:2. He states that oils having similar iodine values but different proportions of component fatty acids may be expected to oxidize at different rates. A direct determination of self-heating properties for any particular oil and potential hazard using the test procedures outlined in this paper (see pages 4-6) is preferable to an empirical evaluation. Even then, since conditions change, it is a good idea to assume that spontaneous combustion could occur and take appropriate precautions.

Bowes (1984) reports that the higher the degree of unsaturation the more tendency the oil has to spontaneously heat. Raw linseed oil is highly unsaturated with over 50% by weight linolenic fatty acids. Boiled linseed oil is not boiled, but has small amounts of driers (e.g., oxides of manganese, lead, or cobalt, or their naphthenates, resinates or linoleates) added to hot linseed oil to accelerate drying. It is possible that these additives increase the potential for spontaneous combustion of boiled linseed oil. Bowes (1984) reported an example of where the amount of material involved in the initiation of a fire could be quite small, as little as 25 grams of rags impregnated with oil. In one series of studies with small samples, 75 grams of cotton rags were impregnated with linseed oil, turpentine and a drying agent of manganese resin ignited in as little as 1 hour to as long as several days. The inconsistency of self-ignition shows that it is wise to use caution whenever dealing with waste products soaked with oil.

CONCLUSIONS

The vegetable oil fuels have a higher propensity for spontaneous combustion than does diesel fuel. Oil soaked rags, waste paper, or porous materials should be handled accordingly. Testing for spontaneous combustion can certainly indicate the existence of a self-heating hazard, but negative results will always remain in doubt. The self-heating process depends on oxidation by atmospheric oxygen at relatively low temperatures, exothermic reactions and the decomposition of unstable substances. Self-heating of rags, sawdust, etc. impregnated with oils can be hazardous if good housekeeping, good maintenance and adequate cleaning

routines are not practiced. Caution should always be used when dealing with oils and rags, sawdust and porous materials. During the testing of boiled linseed oil carried out for this study, some of the heated oil and sawdust mixture was set on a wooden pallet in atmospheric conditions and started a fire. Fortunately, the pallet was outside and the fire was discovered and extinguished before it caused any damage.

Phoenix Chemical Laboratory, Inc., Chicago, Illinois conducts the Differential Mackey Test (ASTM D3523) for a cost of \$125 and up.

RECOMMENDATIONS

Even though both the literature review and the experimental work reported in this paper suggest that spontaneous combustion of sawdust impregnated with the ethyl or methyl esters of rapeseed oil is unlikely, it is still a good idea to take appropriate precautions. Rags, papers or wood particles soaked with Biodiesel should not be left lying in uncovered waste receptacles or allowed to accumulate in corners or on the floor. Storing them in covered waste receptacles and disposing of them promptly is the best way to avoid potential fires. Approved fire extinguisher should be available in areas where these products are used.

Good housekeeping and attention to common fire safety principles is good insurance against fires when using any fuel including Biodiesel

ACKNOWLEDGEMENTS

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The authors also wish to thank the Idaho Department of Water Resources, Energy Division for financial support of this project.

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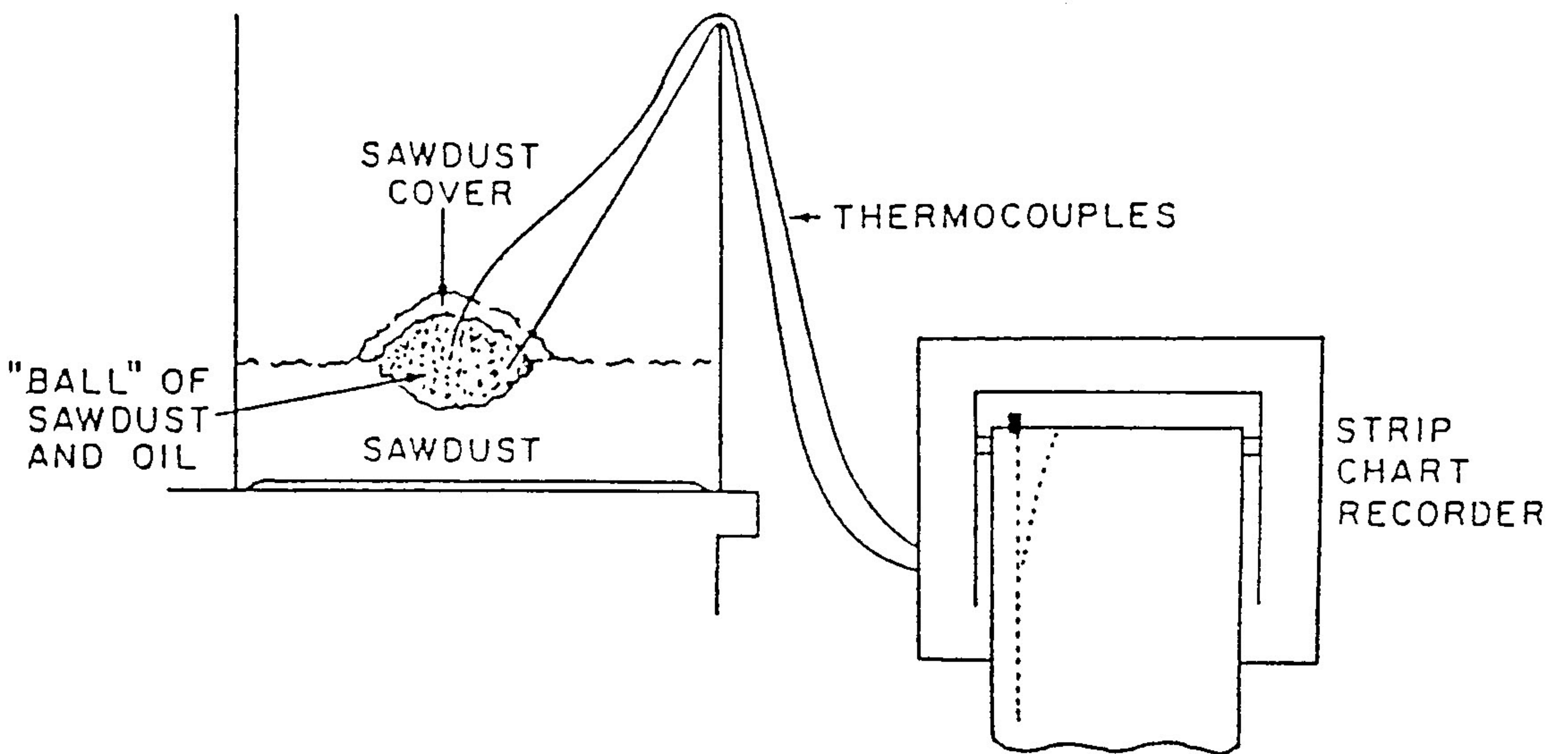


Figure 1. Diagram of Experimental Setup.

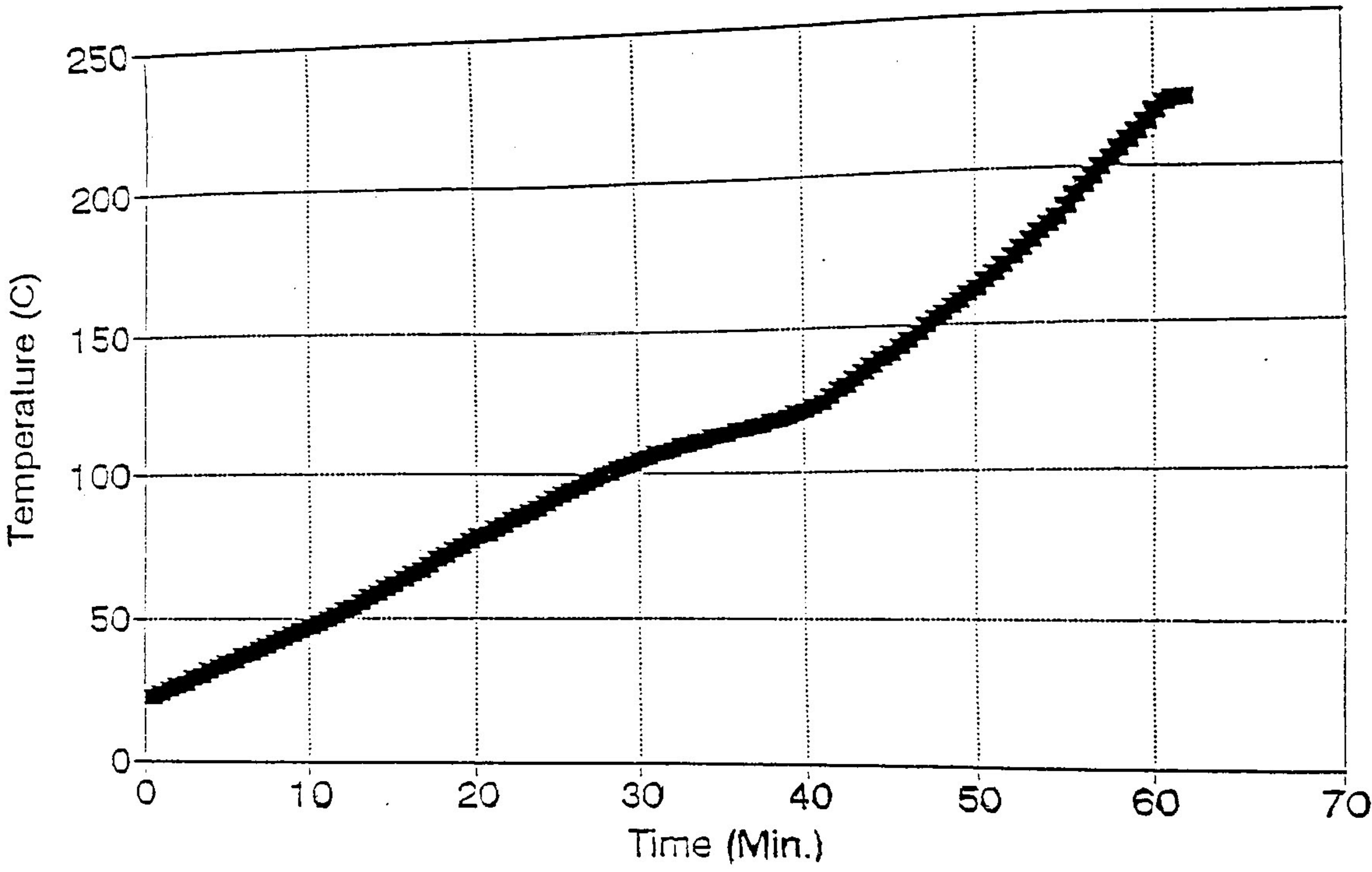


Figure 2. Spontaneous combustion test with 50 grams of linseed oil in 50 grams of sawdust.

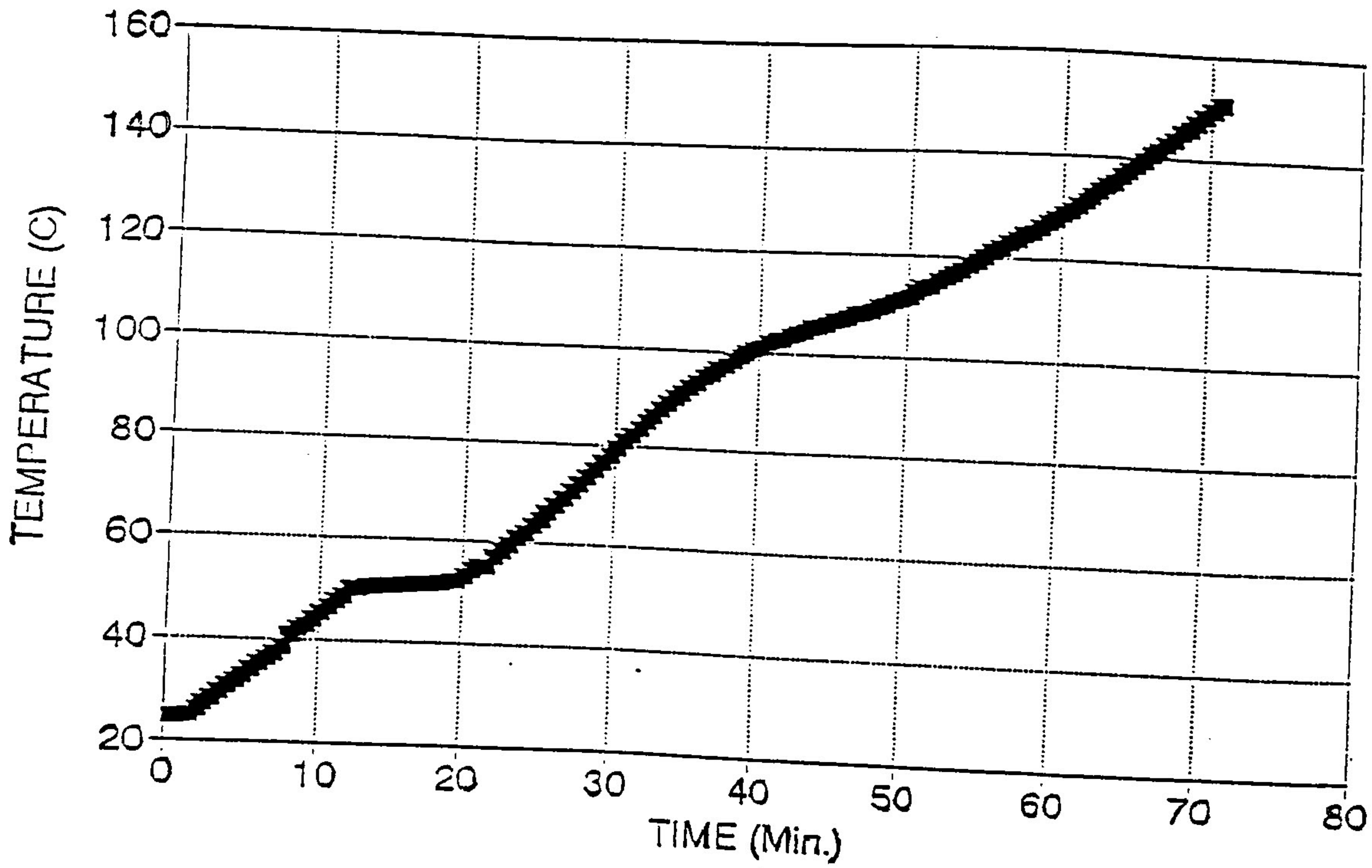


Figure 3. Spontaneous combustion test with 50 grams of RME in 50 grams of sawdust.

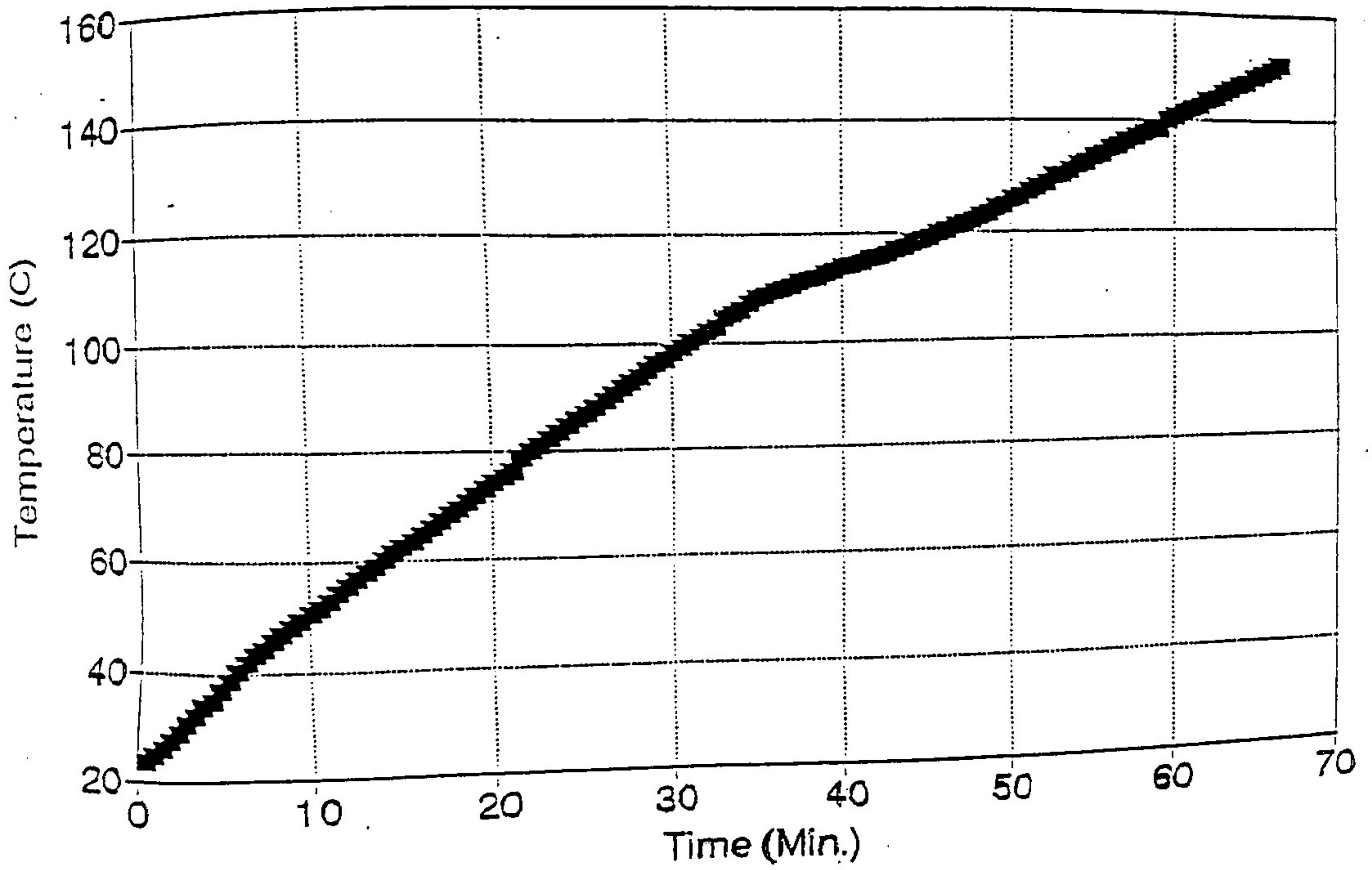


Figure 4. Spontaneous combustion test with 50 grams of REE in 50 grams of sawdust.

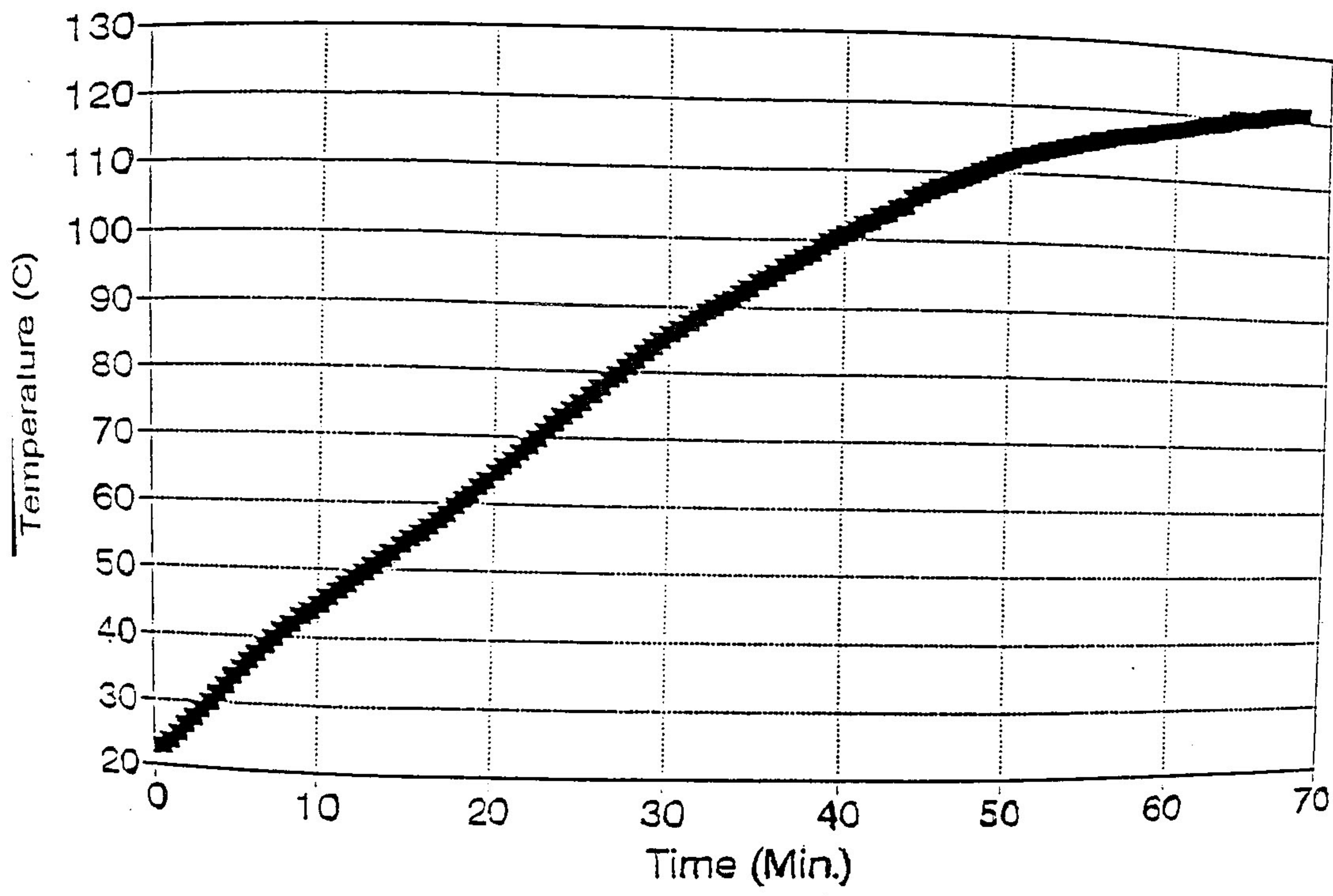


Figure 5. Spontaneous combustion test with a blend of 50 grams of 20% RME - 80% diesel in 50 grams of sawdust.

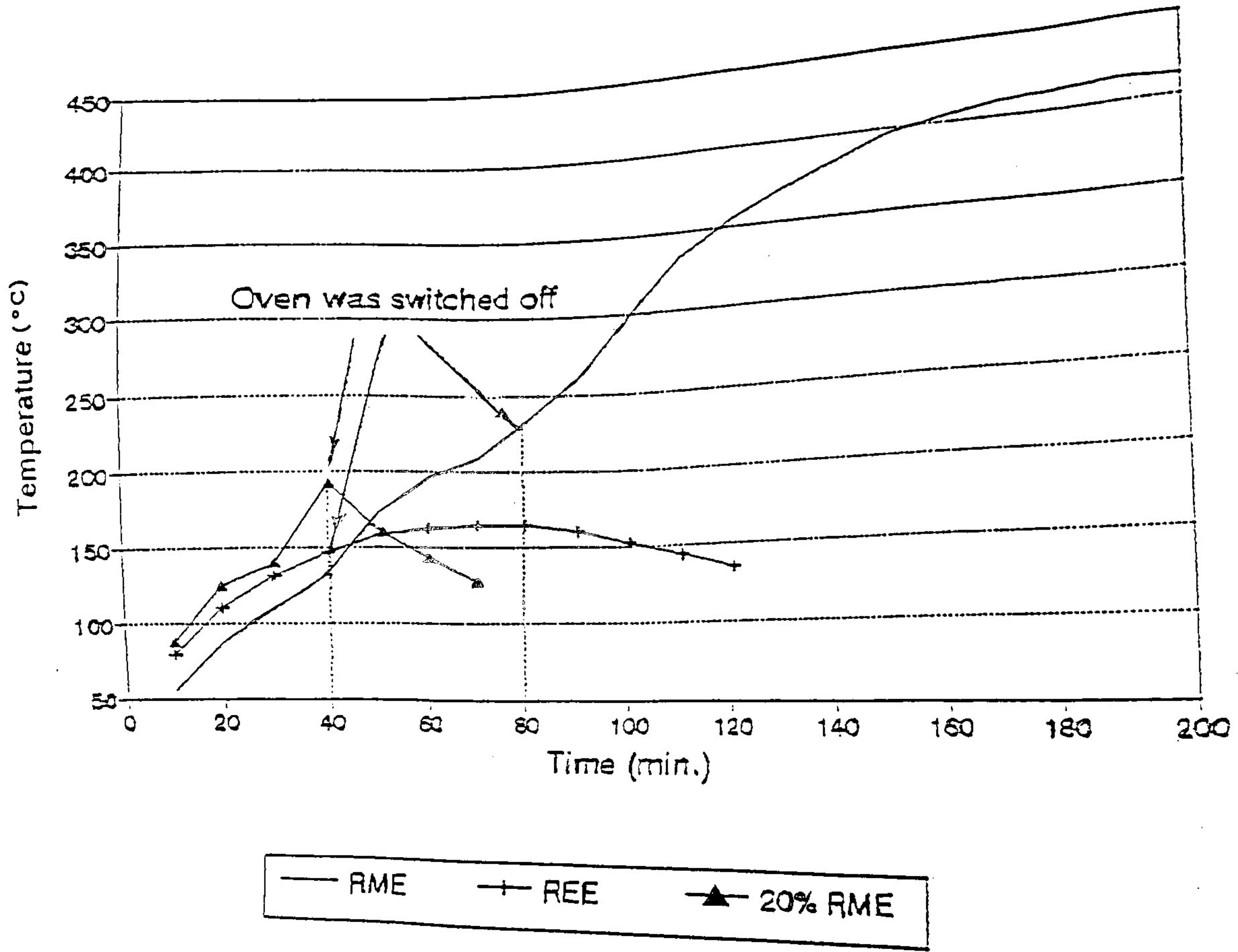


Figure 6. Spontaneous combustion test with RME, REE and 20% RME-80% diesel each in 50 grams of sawdust.