Highly Regulated Power Supply for Hydrogen or Deuterium Arc Lamps Used in Ultraviolet Spectrometry

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In designing detectors for spectrophotometric and fluorometric devices, it became apparent that the power supplies for incandescent and arc lamps generally provided with the older single beam spectrophotometer were not adequate for highly critical work. We are therefore constructing individual circuit modules to provide power for the tungsten and hydrogen lamp sources of a spectrophotometer. The tungsten lamp power supply described in an earlier report provided a constant voltage for a highly stable visible spectrum light output (*1*). In this paper we describe the design and construction of a regulated constant current source and ignition circuit for use with the standard deuterium or hydrogen lamps. The gas discharge tubes used in these instruments consist of a heavy tungsten filament surrounded by a metal tube which, with the filament, serves as the cathode; a few millimeters away is the anode and the light emitting arc is formed between the two electrodes. The lamps depend on thermionic emission from the filament to form and maintain the arc. Initially, arc formation also requires a brief, high potential pulse (several hundred volts) between the electrodes. Once the arc has formed, the interelectrode potential is normally between 75–100 V at the usual current (0.3 A). The supply described here provides the three necessary sources of power to the lamp: (1) low voltage high current (2.5 V, 4 A) ac for the filament, (2) high voltage pulse (800 V for 0.2 ms) for ignition, and (3) the constant current source for sustaining the gas discharge (300 mA at up to 100 V).

CIRCUIT DESCRIPTION AND CONSTRUCTION DETAILS

The hydrogen lamp power supply (Figure 1) will be considered as the three separate functional components described above. The filament is energized by a 2.5-V, 10-A transformer. Originally it had been thought that the filament voltage might need to be regulated either by a constant voltage-harmonic transformer or after rectification by a low voltage dc regulator. In fact, relatively large changes in the filament voltage have little effect on the light output with this supply. A rather high current capacity filament transformer was used to provide a lower operating temperature and thus increased reliability. The center tap of the transformer provided a convenient place to connect the filament to the cathode current sink (transistor Q_3).

The ignition voltage pulse is provided by the relaxation oscillator consisting of unijunction transistor Q_2, resistors R_1 and R_2, and capacitor C_2. The values of R_1 and C_2 together with the high intrinsic standoff ratio of the specified unijunction transistor provide a pulse through the primary of T_4 every 5–10 s. The primary to secondary turns ratio of T_4 increases the amplitude of that pulse to 800 V. The positive component of the pulse passes through D_6 to the anode. The silicon diode (D_6) isolates the secondary of T_4 from the anode supply. As soon as the lamp conducts, transistor Q_1 is turned on by the positive voltage developed across R_{10}. This shuts off the unijunction oscillator and keeps it off while the lamp is on. Transformer T_4 was chosen because it is designed for high voltage across the secondary. It is not certain that other transformers with similar turns ratios would be able to withstand the 800-V pulse. When the supply ignites a hydrogen or deuterium lamp, the output of the high impedance pulse transformer does not rise above the ignition voltage, usually below 350 V. If for any reason the current through the lamp is interrupted, the circuit will automatically reignite the lamp.

The anode current is provided by transformer T_1 (120 V), bridge rectifier (D_{1-4}), choke L_1, and C_1. Pilot lamp (P_{L2}) is mounted on the front panel and is lighted when the discharge lamp is conducting. The pilot lamp bulb chosen is rated at 0.35 A and acts as a fuse in case of a short circuit in the arc lamp housing or wiring. Diode D_5 isolates the anode supply from the ignition pulse source. The use of the filter choke (L) provides a 175-V output prior to ignition. When the lamp begins to conduct, the voltage drops to 105 V. This provides a high voltage at the time of ignition to ensure a reliable start and then the lowered output voltage reduces dissipation in the series pass transistor Q_3. Neon lamp P_{L3}, together with resistor R_8, provides protection of transistor Q_3 against high voltage transients, especially the ignition pulse. The neon lamp fires at 80–90 V and thus prevents the voltage on the collector of Q_3 from rising substantially above that level for short-lived transients.

Regulation of the arc current through the lamp is provided by a constant current sink which senses changes in current by comparing the voltage across resistor R_{10} with a reference voltage provided by diode D_9 and resistor R_5 (*2*). The operational amplifier IC_2 compares the two potentials and, as appropriate, provides more or less drive current to pass transistor Q_3. This negative feedback loop with a gain of about 10^5 ensures a constant potential across R_{10} (3 V) and thus a constant current through the discharge lamp which is in series with it. The major contribution to the small current variations with time are due to temperature induced changes in the offset voltage of the integrated circuit amplifier, the resistors, and reference diode D_9. For that reason it is essential that resistors R_4, R_5, R_6, R_7, R_9, and R_{10} have low temperature coefficients (less than 100 ppm/°C) and that diode D_9 be a temperature compensated reference diode rather than a simple zener diode. Transformer T_3, diodes D_7 and D_8, capacitor C_3, and voltage regulator IC_1 provide a constant voltage source (+15 V) for operation of the relaxation oscillator, reference diode, and operational amplifier. The last is specified to be an RCA CA3140 integrated circuit which, unlike most operational amplifiers, requires only one power supply (*3*). Better stability might be obtained with an operational amplifier having a lower offset voltage temperature coefficient, but that would require the addition of a −15-V supply.

The power supply was assembled using standard construction practices and enclosed in a 6 × 10 × 7 inch aluminum box. The components shown within the dotted line on the schematic diagram were mounted on a 3 × 5 inch printed circuit board (the printed circuit board layout pattern is available from the author on request). The components could as well have been assembled on a piece of Vectorboard or similar substrate. In that case, special attention should be given to short lead lengths to avoid instability in the feedback loop. All ground leads were brought to one point on the cabinet. Transistor Q_3 was mounted on the specified circuit board heat sink with a thin film of silicone grease to improve heat conduction. Transformers T_1 and T_2, as specified, have the same spacing of their mounting holes and were stacked to provide for a more compact assembly.

Four screen-covered $^3/_4$-in. holes were placed at each end of the cabinet for ventilation. The circuit board was positioned to allow R_5 to be adjusted through one of them. Jacks were provided on the back of the cabinet to allow easy measurement of the voltage across R_{10}. The cathode and anode leads for the gas discharge lamp were brought to a female socket on the back of the supply which mates with the socket on the cable from the spectrophotometer lamp housing.

Prior to attaching the supply to the lamp with which it is to be used, a 100–300 Ω 50-W resistor should be inserted between the anode output and one of the cathode sink terminals. The supply can be adjusted with R_5 to give 3 V across R_{10} indicating

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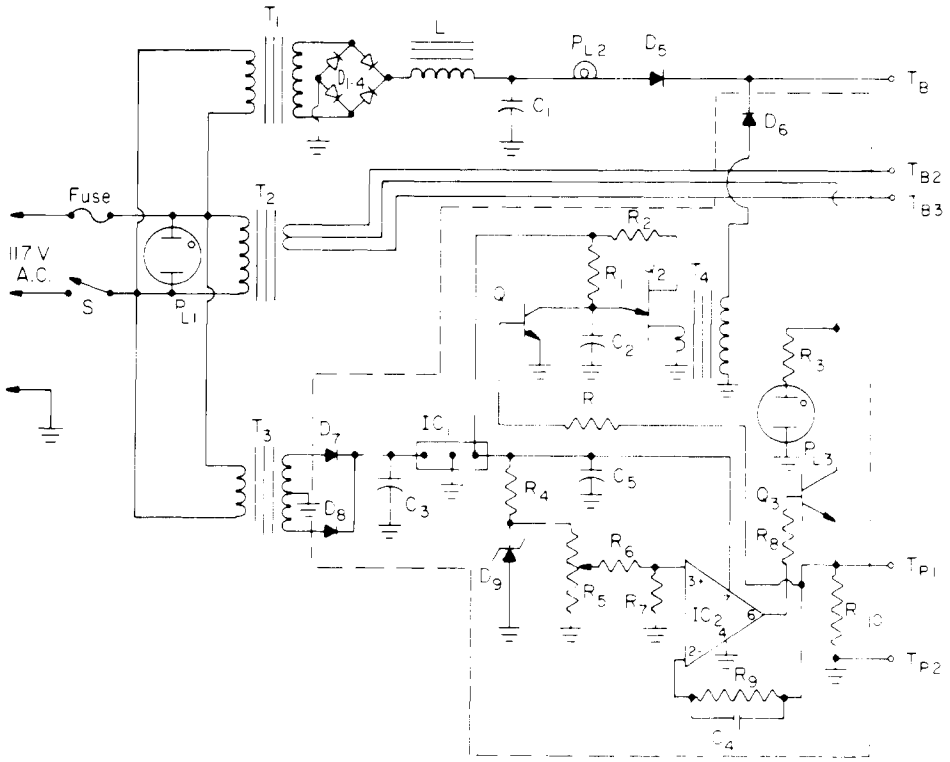

Figure 1. Schematic diagram of hydrogen lamp power supply. Component list: Q_1, 2N6175 transistor; Q_2, 2N4871 transistor; Q_3, MJ410 transistor (Motorola); D_1 through D_8, 1N4007 rectifier diodes; D_9, 1N938B temperature compensated reference diode (9 V); IC_1, μA7815, +15-V regulator (Fairchild); IC_2 CA3140T operational amplifier (RCA); C_1, 200-μF, 250-V electrolytic capacitor; C_2, 47-μF, 25-V electrolytic capacitor; C_3, 100-μF, 50-V electrolytic capacitor; C_4 and C_5, 0.047-μF, 100-V Mylar capacitor. R_1, 47-kΩ, 1/2-W carbon resistor; R_2, 100-Ω, 1/2-W carbon resistor; R_3 and R_{11} 27-kΩ, 1/2-W carbon resistor; R_4, 825-Ω, 1/2-W metal film resistor; R_5, 10-kΩ Cermet 10-turn trim potentiometer; R_6 and R_7, 17.8-kΩ, 1/2-W metal film resistor; R_8, 680-Ω, 1/2-W carbon resistor; R_9, 2.3-kΩ, 1/2-W metal film resistor; R_{10}, 10-Ω, 5-W wire wound resistor; T_1, 120-V, 0.5-A transformer (Signal Transformer Co., N.Y., Model 241-7-120); T_2, 2.5-V 10-A transformer (Triad F-3X); T_3, 28-V, C.T., 0.08-A transformer (Signal 241-3-28); T_4, blocking oscillator transformer (Triad TY-202X); L, 0.8-H, 375-mA, 25-Ω inductor (Stancor C2328); P_{L1}, neon pilot lamp assembly for 117 V; P_{L2}, 3.2-V, 0.35-A bulb (General Electric No. 45); P_{L3}, Neon Lamp (NE-2H) Fuse; 2-A, 250-V

a current of 300 mA. A 117-V, 60-W incandescent light bulb should serve well for such a dummy load. Final adjustment in current can be made after the supply has been in operation with the hydrogen or deuterium lamp. The lamp anode connected to T_{B1} and the filament leads to T_{B2} and T_{B3}.

OPERATING CHARACTERISTICS

The supply has been in use for 13 months with four different deuterium lamps. Three of them were ignited by the first pulse after the 7-s warmup. The fourth ignited prior to the pulse by the 175-V output of the anode supply under no load conditions. The current stability of the supply was assessed by using power resistors as dummy loads. The resistance ranged from 100 to 275 Ω. Current was monitored by measuring the voltage drop across a 0.6-Ω thermostated resistor with a four digit voltmeter. Beginning 10 s after the supply was turned on, the current changed less than 1% over a 3-h period. After a 10-min warmup of the supply, changes in all cases were less than 0.1% over 3 h. The change in current as a function of change in load resistance was determined to be 0.013 mA/Ω. Additionally, the stability of the supply was determined as a function of changes in light output with time. The supply was attached to the deuterium lamp of a Beckman DU spectrophotometer. The standard Beckman supplied photomultiplier tube and associated electronics were used as a detector. Measurements were made at both 260 and 205 nm in separate experiments. With the monochromater exit slit set at 0.02 mm for 260 nm and 0.4 mm for 205 nm, the sensitivity of the photomultiplier was adjusted to give an apparent absorbance of 0.200 with no sample in the light path. The deuterium lamp and all electronics except the supply under test were operated 2 h by means of an independent power source in order to stabilize both the detector system and any thermally-induced mechanical changes in the lamp housing and focus system. Measurements of the apparent absorbance were made over a 3-h period beginning 30 s after the supply under test was turned on. At 260 nm, the apparent absorbance at all times was 0.200 ± 0.003. After 10 min, the total short-term drift was less than 0.001 during any 20-min interval. For the 205-nm wavelength, the maximum deviation from an absorbance of 0.200 was ±0.005 with a short-term drift of 0.003/20 min. Starting with a cold lamp and housing, about 30 min were required to achieve similar stability in the apparent absorbance. The drift during that period was probably due to the mechanical changes in the lamp and its housing. Changes in the nominal 117-V ac line voltage between 110 and 130 V caused a variation of less than ±0.002 absorbance unit; from 95 to 140 V input, the changes were ±0.004. Since the filament voltage on the arc lamp is unregulated, it changes in proportion to the input voltage; the highly regulated anode current must prevent the changes in light output which would otherwise occur with such large differences in filament temperature. The power supply described here seems to be close to reaching the limit of useful stability with lamp supplies for ordinary single beam, general use laboratory spectrophotometers. Heat induced mechanical changes in the optical system and drift in the detector become important in trying to increase the stability. Obviously the use of double beam or other self-compensating spectrophotometers is to be recommended when greater freedom from drift is needed. Total costs of materials, if purchased in 1977 in unit quantities, would be $80 including a coat of epoxy paint on the cabinet. The supply has been in use for a year without problem. As with the previously published design for the tungsten lamp supply, it is very important to use high quality, conservatively

rated components to increase the operating time before breakdown (*1*).

LITERATURE CITED

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Carboxylate Anions from Fatty Acids by Esterification with *p*-Nitrobenzyl Bromide for Analysis of Fatty Acid Mixtures by Negative Ion Mass Spectrometry

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In the analysis of fatty acids, often the problem is with the separation, since they usually exist as complex mixtures in natural products such as fat or lipids. Although it is relatively straightforward to separate fatty acid mixtures composed of homologues by GC or LC, the complete separation of fatty acids from natural products is sometimes very difficult, because they contain various types of fatty acids with or without double bonds.

Negative ion mass spectrometry, which usually yields simple spectra highly characteristic of the types of compounds, has been applied to the analysis of alcohols and aldehyde mixtures (*1*), and mercaptans (*2*).

This report describes the qualitative analysis of fatty acid mixtures by negative ion mass spectrometry. The negative ion mass spectra of fatty acid *p*-nitrobenzyl esters yield carboxylate anions as the base peak, and molecular anions and other fragments become negligibly small. By this derivatization, more than several hundreds times enhancement is achieved on the carboxylate anions.

EXPERIMENTAL

Reagents. 18-Crown-6(1,4,7,11,13,16-hexaoxycyclooctadecane) was purchased from Nippon Soda Company, Japan. *p*-Nitrobenzyl bromide and phenacylbromide were purchased from Tokyo Kasei Industries, Japan, and recrystallized from methanol/water solutions. The stock benzene solutions of *p*-nitrobenzyl bromide (22 mg/mL) and phenacylbromide (20 mg/mL) were prepared, where the molar ratio of alkylating reagent/crown ether was adjusted to 20:1, respectively.

Alkylating Procedure. Alkylation was performed according to the method by Durst et al. (*3*). A fatty acid sample (ca. 0.5 mM) was dissolved in 2 mL methanol and neutralized to a phenolphthalein end point by a KOH/methanol solution (0.5 N). After the solution was evacuated to remove the solvent by rotary pump, 5 mL of the alkylating solution was added and the suspending solution was refluxed for 30 min to complete the reaction. The resulting solution was used for mass spectrometric measurement.

An oil sample about 80 mg(0.4–0.3 mM of total fatty acids) was dissolved in 4 mL of the KOH/methanol solution. After cooling, the solution was neutralized to a phenolphthalein end point by a HCl/methanol solution (0.5 N). The following procedures are the same as mentioned above.

Apparatus. JMS-01SG double focusing mass spectrometer from JEOLCO was used in the negative ion mode under the following conditions: ionizing energy of 150 eV, ionizing current of 200 μA, accelerating voltage of 6 kV, ion source temperature of 130–200 °C and ion source pressure of 10^{-5} Torr. All mass spectra were obtained using a direct inlet probe.

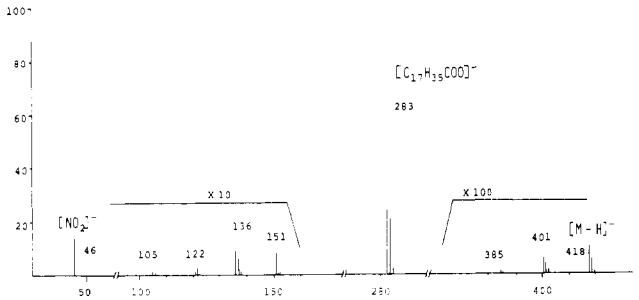

Figure 1. Negative ion mass spectrum of *p*-nitrobenzyl stearate

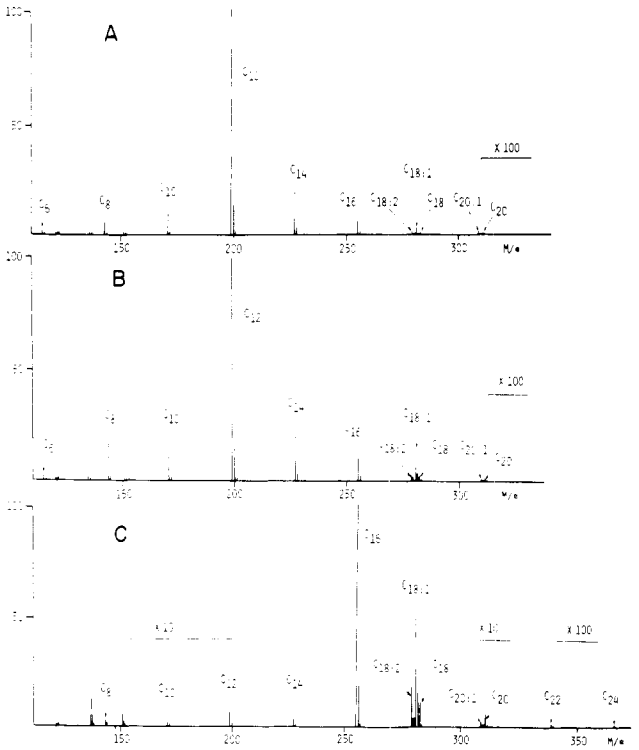

Figure 2. Negative ion mass spectra of *p*-nitrobenzyl esters of fatty acids from (A) coconut oil, (B) palmseed oil, and (C) palm oil

RESULTS AND DISCUSSION

Figure 1 shows the negative ion mass spectrum of *p*-nitrobenzyl stearate. The other *p*-nitrobenzyl esters show basically the same type of spectra. The peak of $[RCOO]^-$,

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